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REPORT

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October 12, 1973

ACID ELECTROLYTE FUEL CELL TECHNOLOGY PROGRAM

FINAL REPORT

Contract NAS 9-12332
Data Item No. 9

Prepared for
National Aeronautics and Space Administration
Lyndon B. Johnson Space Center
R and D Procurement Branch
Houston, Texas 77058

DIRECT ENERGY CONVERSION PROGRAMS

AIRCRAFT EQUIPMENT DIVISION
LYNN, MASSACHUSETTS

GENERAL ELECTRIC

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ACKNOWLEDGEMENTS

This final report summarizes the Acid Electrolyte Fuel Cell Technology Program conducted by the General Electric Company, Direct Energy Conversion Programs, for the National Aeronautics and Space Administration under Contract NAS 9-12332. The period of performance was October 1, 1971 through May 15, 1973.

This work was performed under the guidance of Mr. G. D. Hydrick, Jr., Program Manager for the Power and Propulsion Branch of NASA/Lyndon B. Johnson Space Center, Houston, Texas. The overall program was directed by Mr. L. E. Chapman, Program Manager, GE/Direct Energy Conversion Programs, Lynn, Massachusetts.

1.0 OBJECTIVE

The objective of this program was to advance the technology of the acid electrolyte fuel cell (solid polymer electrolyte) for a cost-effective electrical power system for the Space Shuttle Orbiter and other future spacecraft.

The earlier work performed by General Electric under Contract NAS 9-11033 and described in Final Report SPR-044, dated 25 September 1971, identified the configuration and operating parameters which were life limiting in the original baseline design. The work performed under Contract NAS 9-11876 and described in Final Report SPR-060, dated 30 November 1971, demonstrated a breakthrough in the life capability of this hardware by proper prehumidification of the reactant gases.

The objective of this present contract was to complete the development and evaluation of flightweight design concepts for the integration of the reactant prehumidification into the cell or stack configuration. The end product of this program was the fabrication and 2000-hour life test of a prototype Engineering Module, integrating the developed product improvements into the baseline cell assembly configuration with the ancillary components of a Fuel Cell Power Supply Module.

2.0 BACKGROUND

Development efforts under the previous contract had established clearly the requirement to humidify the reactant gases before entering the active area of the cell in order to prevent localized drying and the consequent stressing of the solid polymer electrolyte (SPE). A significant reduction of the fluoride ion level in the product water was demonstrated with the incorporation of a hydrogen peroxide scavenger in the SPE when operated in conjunction with the prehumidified reactants. It had been determined that a performance loss of 30-40 millivolts per cell occurred in the bonded cell assembly configurations when compared with gasketed boilerplate hardware configurations. This loss was attributed to a contaminating constituent in the AF-42 bonding adhesive which affected the activity of the cathode catalyst. It was decided to evaluate possible alternate adhesives.

The approaches taken under this contract in these areas of technology development are described in the following section.

3.0 APPROACH

3.1 Reactant Prehumidification

There were two alternative approaches studied and evaluated for accomplishing the prehumidification of the reactants in a flight configuration fuel cell system. One approach was to prehumidify the reactants before entry into the stack assembly. The other approach was to humidify the reactants at the entry to each individual cell assembly. Both of these approaches were evaluated in small stack buildups by life testing.

The concept of prehumidifying the reactants before entry into the stack assembly was demonstrated in breadboard hardware during Contract NAS9-11033. This is the subject of a patent docket issued to Mr. D. W. Craft of the General Electric Company, and is reported under the New Technology Clause of the contract. The design approach is to use the SPE as a liquid/gas separator with the product water passing by one side and the incoming reactant passing by the other side. See Figure 1. The coolant from the exit side of the stack is passed through the humidifier to provide the heat of vaporization and to condition the reactants such that they enter the cell assemblies saturated at a temperature slightly above the cell temperature at the reactant and coolant inlet side of the cell assembly. Breadboard models of this concept were tested for 900 hours and provided design data on water transport rates, gas flow rates and heat transfer. The approach to this contractual effort was to design a flightworthy configuration of the concept that would be integrated with the stack assembly end plate. Following component evaluation tests, this prehumidifier package was assembled into the full stack assemblies used in the Engineering Model life tests.

The alternate humidification concept was to provide available product water from the cathode-side wicks to the reactant inlet areas of each cell to humidify the gases before they contacted the catalyzed active areas. See Figure 2. This approach was fabricated into the hydrogen inlet area of a small stack buildup (B/U 102) under the previous contract. The unit operated for 1762 hours until failure, which was caused by loss of the "prime" in the wicking system following a facility failure. This approach is considered as the most direct solution, but results in a reduction of 7 - 10% of the current active cell area.

3.2 Hydrogen Peroxide Scavenger

The laboratory matrix testing of the operational and configuration parameters affecting cell life indicated a significant advantage to the use of a hydrogen peroxide scavenger in the SPE in conjunction with the prehumidified reactants. A process had been developed under prior development programs to infuse platinum particles into the SPE to act as a scavenger for hydrogen peroxide formed internal to SPE from the reactions of the hydrogen and oxygen diffusing through the polymer.

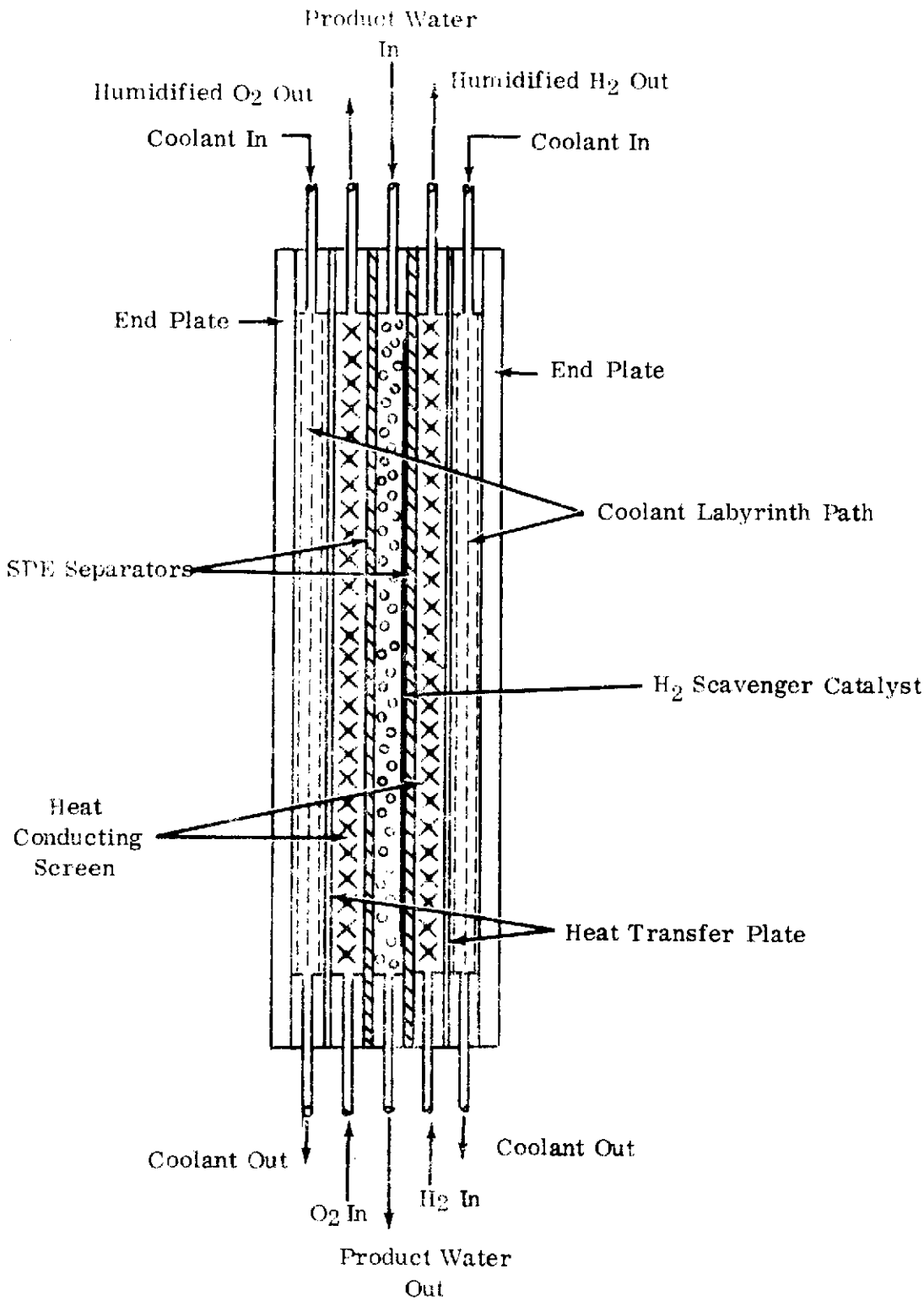


Figure 1. Reactant Prehumidifier Schematic

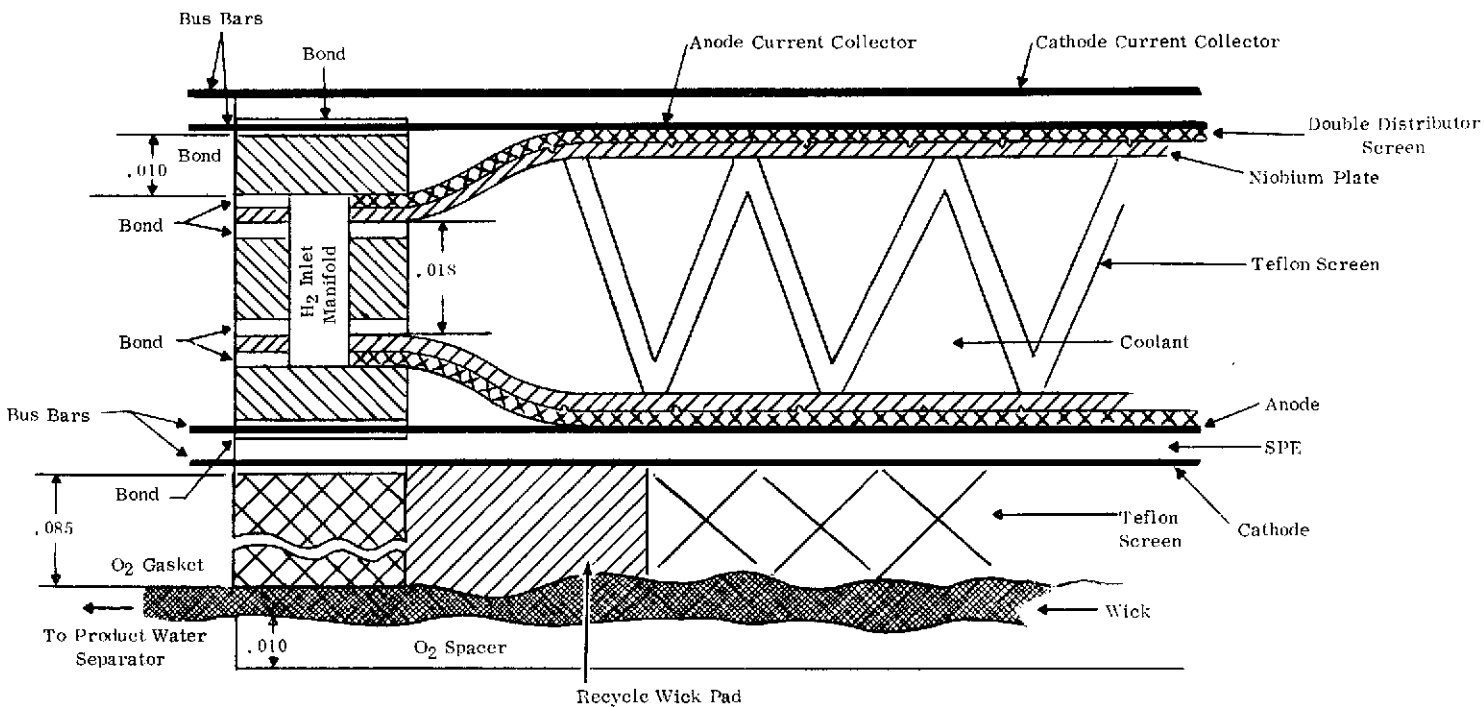


Figure 2. Cell Wick Pad Prehumidifier Schematic

Therefore, the approach taken in this program was to evaluate a small stack buildup with platinized SPE's operating at 180°F and 60 psia with prehumidified reactants.

3.3 Performance

The task of identifying the constituent in the AF-42 bonding adhesive which caused the cathode contamination was pursued by NASA/JSC. Other similar adhesive systems which did not contain the contaminant were also evaluated by NASA/JSC. This investigation was performed by Dr. M. Clark, under the direction of Mr. Hoyt McBryer. The results are contained in a report dated March 21, 1972. Traces of cyanide were found evolving from the AF-42 adhesive, which was believed to be the contaminating constituent. A comparable bonding material produced by American Cyanamid designated FM-1000 was found to be free of this constituent. The approach included the fabrication and test evaluation of a small stack buildup containing an alternate bonding material.

3.4 Engineering Module

The conclusion of this program was planned to include the fabrication of two full stacks incorporating the integrated prehumidifier features as well as the proven configuration changes from the prior small stack tests. These stacks were to be assembled into a prototype of the container and structures assembly plus the ancillary components considered as an integral part of the Fuel Cell Power Supply Module as it would apply to the Space Shuttle Orbiter. The only exceptions to the development of flight-type ancillary components were the reactant pressure regulators and the prototype packaging of the electronic monitoring and control unit. The reactant pressure regulators originally procured and evaluated under the previous contracts were designed for use with low-pressure gas supplies and were not adequate when the Space Shuttle baseline settled on dedicated supplies of supercritical cryogenic reactant storage. Since regulators for this type of use were considered as state-of-the-art technology, it was concluded that it was not necessary to spend technology development funds to tailor regulators for this specific application. The electronic circuitry for completely automatic monitoring and control of the fuel cell module had been developed in a breadboard arrangement and used as part of the test facilities. However, it was considered unnecessary to develop a prototype packaging of this component for attachment to the module.

4.0 Technology Activities

4.1 Small Stack Testing

4.1.1 B/U 101A

The original small stack buildup was fabricated and operated successfully for 2000 hours under Contract NAS9-11896. Figure 3 shows this unit in a test configuration. At the 2000-hour point, one cell assembly was removed from the stack for complete chemical and physical analysis of the solid polymer electrolyte. The cell assemblies in this unit were non-platinized SPE's and were operated at 150°F and 45 psia with the reactants prehumidified in the test facility. The analysis of the cell assembly showed essentially no change in physical or chemical characteristics from a new cell.

The unit was reassembled as a three-cell assembly stack with new gaskets designed to improve the support to the bonded area of the assembly. A shut-off valve was installed in the facilities tied into the automatic shutdown control to isolate the product water system in the event of a unit or facility failure to prevent the loss of prime in the wicks as had occurred in the life test of B/U 102.

The buildup was operated for another 3000 hours at the 150°F and 45 psia operating conditions, accumulating a total of 35 simulated missions using the electrolysis startup method. At this point, the water separator was replaced and the testing continued for another 1500 hours, using the vacuum startup method. The operating conditions were 180°F and 45 psia from the 5700-hour point to the termination of the test at 6500 hours.

The operation of this small stack buildup for a total of 6500 hours demonstrated the very long life with invariant performance capabilities inherent in the SPE when operated with prehumidified reactants. It also demonstrated the integrity of the baseline cell assembly hardware when provided with the proper backup support to the bonded joints. A total of 43 mission cycles were successfully completed, demonstrating both the electrolysis and vacuum startup procedures. The chronological plot of the performance at the baseline load conditions is shown in Figure 4. The polarization data at the various operating temperatures and pressures evaluated is shown on Figure 5.

The test report for Small Stack Buildup No. 101A is GE/DECP publication SPR-053, Supplement No. 1, dated 10 October 1972.

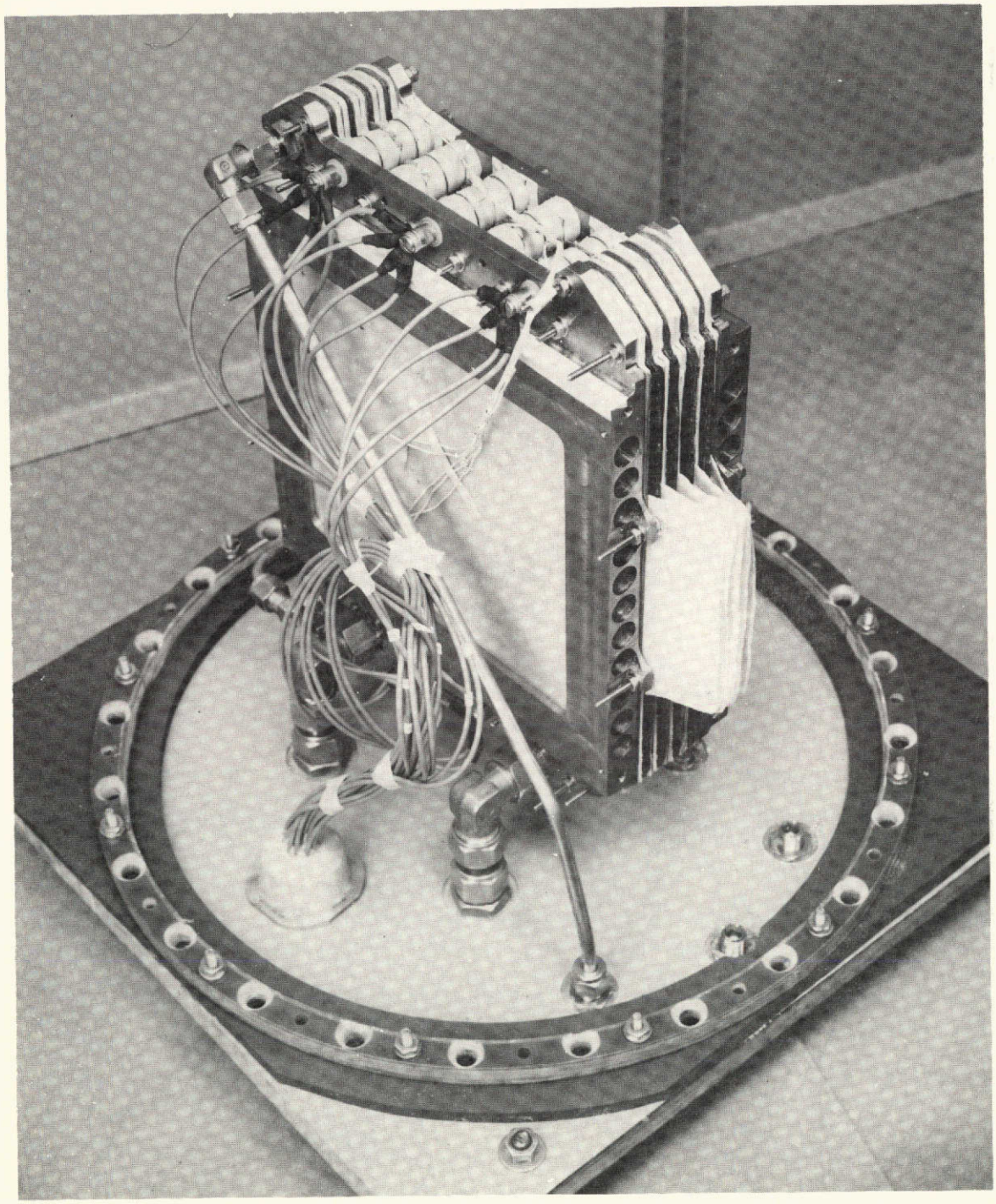


Figure 3. Configuration of Small Stack Test Buildups

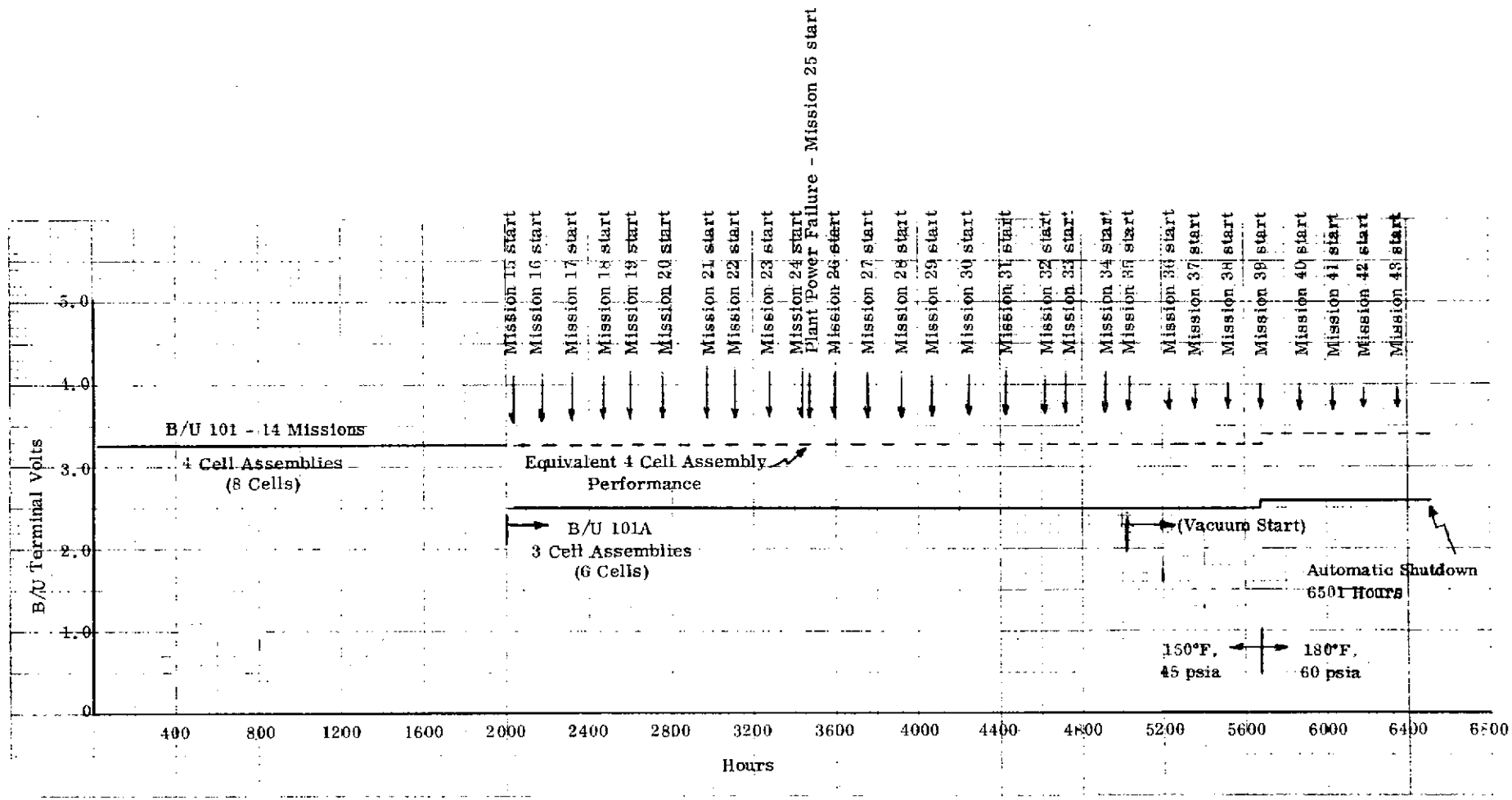


Figure 4. B/U 101 Performance at 125 Watts Output

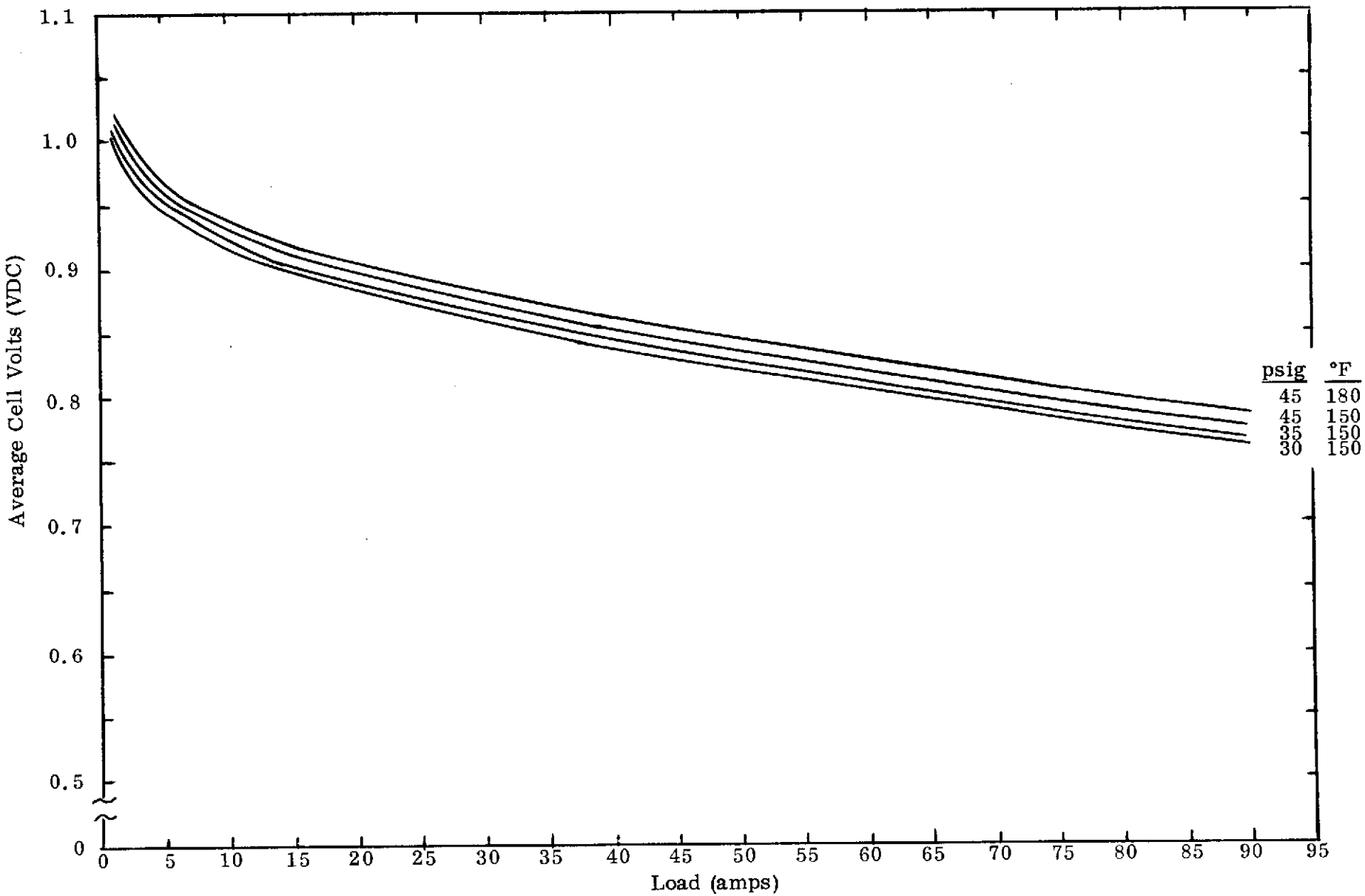


Figure 5. B/U 101B Polarization Data

4.1.2 B/U 102

This small stack buildup of four cell assemblies was fabricated and tested under Contract NAS9-11876. The cell assembly configuration was exactly the same as B/U 101 except for the placement of the internal recycle wick pad on an uncatalyzed area of the hydrogen inlet. The unit was operated at 150°F and 45 psia without the external prehumidification in the facilities. Figure 6 shows B/U 102 test performance.

Although the unit failed after 1762 hours of operation due to a test facilities related breakdown, the laboratory analysis of the cells showed no signs of polymer degradation at the bottom of the cell where the recycle wick kept the cell inlet area saturated. A facility power failure caused the test unit to be shut down, leaving the product water line open with a ΔP across the water separator, while no water was being generated by the cell assembly. This caused the wick pad at the top hydrogen inlet area to lose its prime. When the unit was restarted, that area operated with dry gases entering the cell. This resulted in the same type of polymer degradation occurring in that area as had been experienced in the prior hardware testing. Even though this test indicated a promising evaluation of the internal hydrogen humidification concept, this approach was abandoned in view of the overwhelming success of B/U 101 with prehumidification of both reactants prior to entry into the stack and the successful evaluation of the integrated prehumidifier as a separate component.

The test report for Small Stack Buildup No. 102 is GE/DECP publication SPR-054, dated 3 November 1971.

4.1.3 B/U 103

This small stack buildup consisted of four cell assemblies fabricated to the same configuration as B/U 101 and 102. The unit was operated at 180°F and 50 psia to evaluate the performance and life capability of the baseline configuration when operated at the higher temperature in conjunction with the prehumidified reactants. See Figure 7. The unit failed after 599 hours of operation, due to a human error in the conduct of the test. The heater setting on the oxygen prehumidifier control was set too high, resulting in reactant temperatures entering the cells at more than 250°F. The teardown evidence indicated that the failure resulted from an oxygen-to-hydrogen leakage occurring upstream of the SPE assembly. It is suspected to have occurred at the hydrogen manifold between the gasket and the cell assembly ears, which were distorted by the overtemperature condition.

The test report for Small Stack Buildup No. 103 is GE/DECP publication SPR-077, dated 15 May 1972.

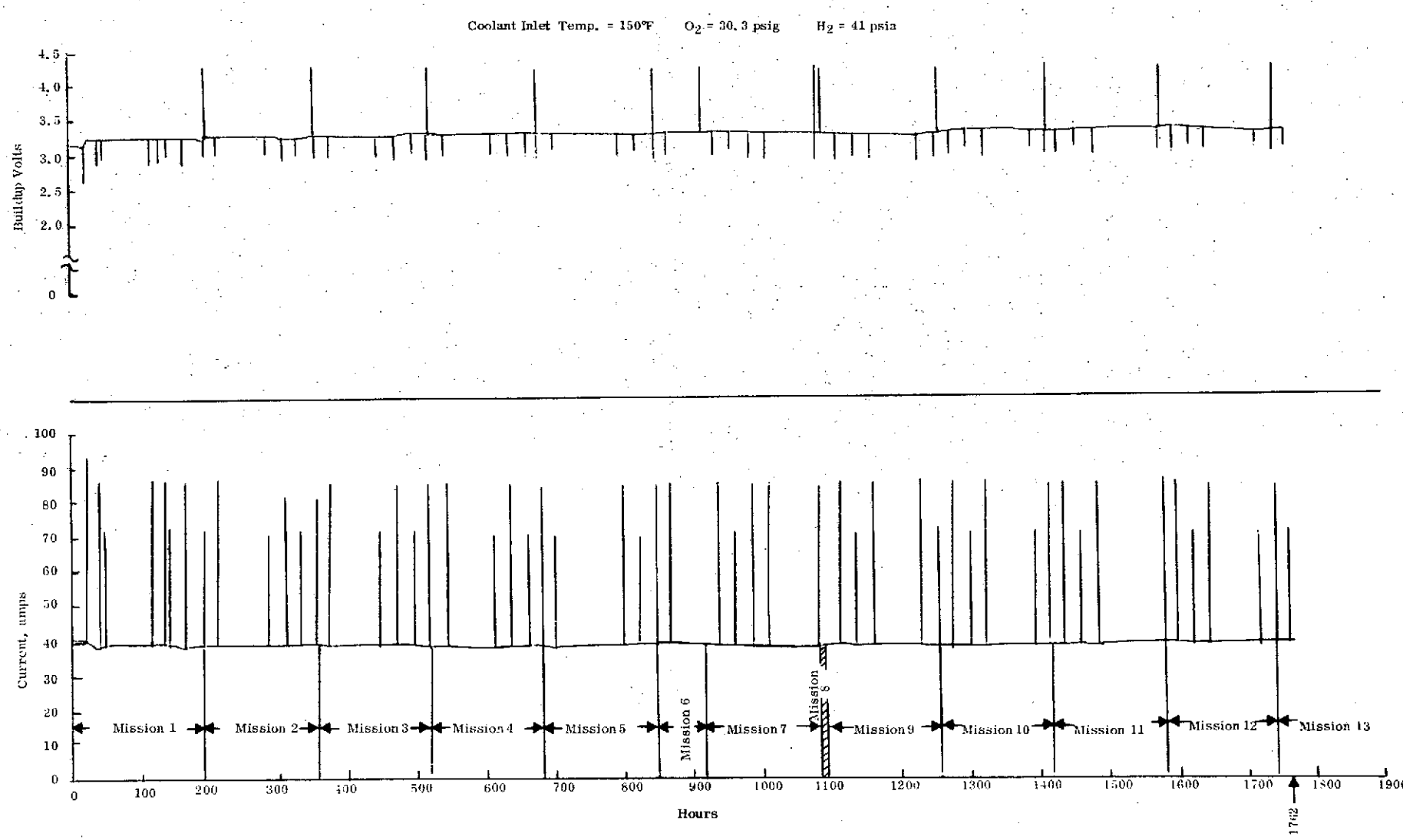


Figure 6. B/U 102 (4 cells) Test Performance

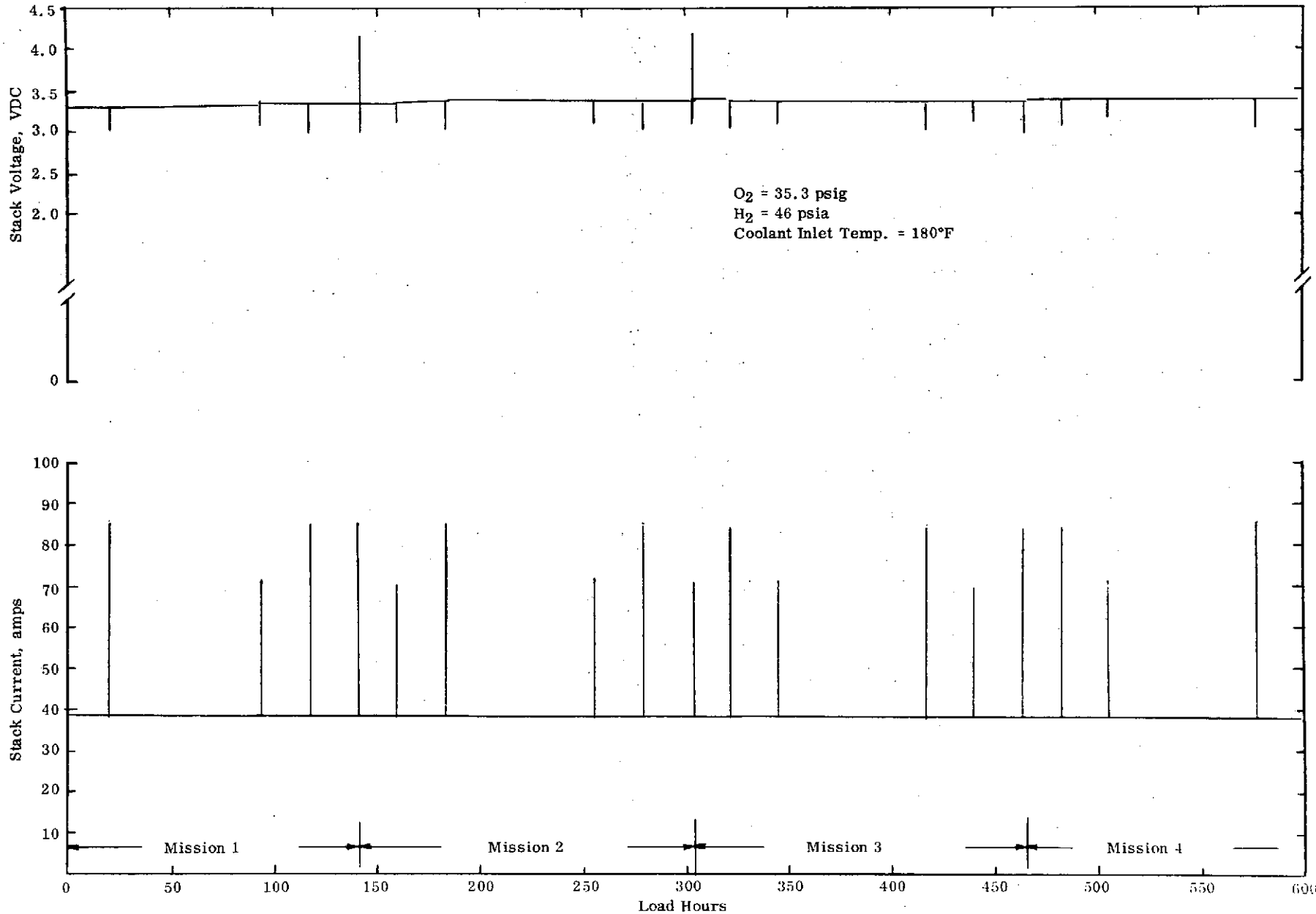


Figure 7. B/U 103 (4-Cells) Test Performance

4.1.4 B/U 104

This small stack buildup consisted of four cell assemblies fabricated to the same configuration as B/U 101 and 102, except that the SPE was platinized to provide a scavenger for hydrogen peroxide. The laboratory matrix of tests had indicated a significant reduction in the fluoride ion in the product water when the platinized SPE was used in conjunction with prehumidified reactants.

The unit was operated for the first 1200 hours at 150°F and 45 psia in order to establish a correlation of fluoride ion count with B/U 101 under the identical operating conditions. Where analysis of the product water from B/U 101 showed 6 to 8 ppm of fluoride ion, the water from B/U 104 showed less than 1 ppm. For the next 3124 hours of operation, the temperature was increased to 180°F, and after the 2100-hour point, the oxygen pressure was increased to 60 psia.

For the first 2600 hours and 18 simulated missions, the electrolysis method of startup was used. The vacuum startup procedure was used for the following 1724 hours and 10 simulated missions.

During the entire operation of B/U 104, the fluoride level in the product water remained at less than 1 ppm, which was the minimum level of sensitivity of the measuring instrument. The chronological plot of the performance at the baseline load condition is shown on Figure 8. The polarization data at the various operating conditions is shown on Figure 9.

The cause for termination of the B/U 104 test after 4324 hours of operation was excessive O_2-to-H_2 leakage, which resulted in an automatic shutdown of the unit. The teardown analysis revealed a crack in the 20-mil thick polysulfone frame between the cooling cartridge and the SPE. From the impression of the crack in the SPE, it was concluded that the frame was probably cracked during fabrication. However, it remained sealed by the flash from the bonding material on either side until such time as the exposed bond material deteriorated to the point where it would no longer seal the ends of the crack.

The test report for Small Stack Buildup No. 104 is GE/DECP publication SPR-094, dated 28 November 1972.

4.1.5 B/U 105

This small stack buildup of four cells was assembled in October 1972. The cells were bonded with FM-1000 adhesive in an effort to eliminate the catalyst contamination encountered with the AF-42 adhesive. Testing by NASA/JSC indicated that the FM-1000 was non-contaminating and that the strength vs. life in 200°F water

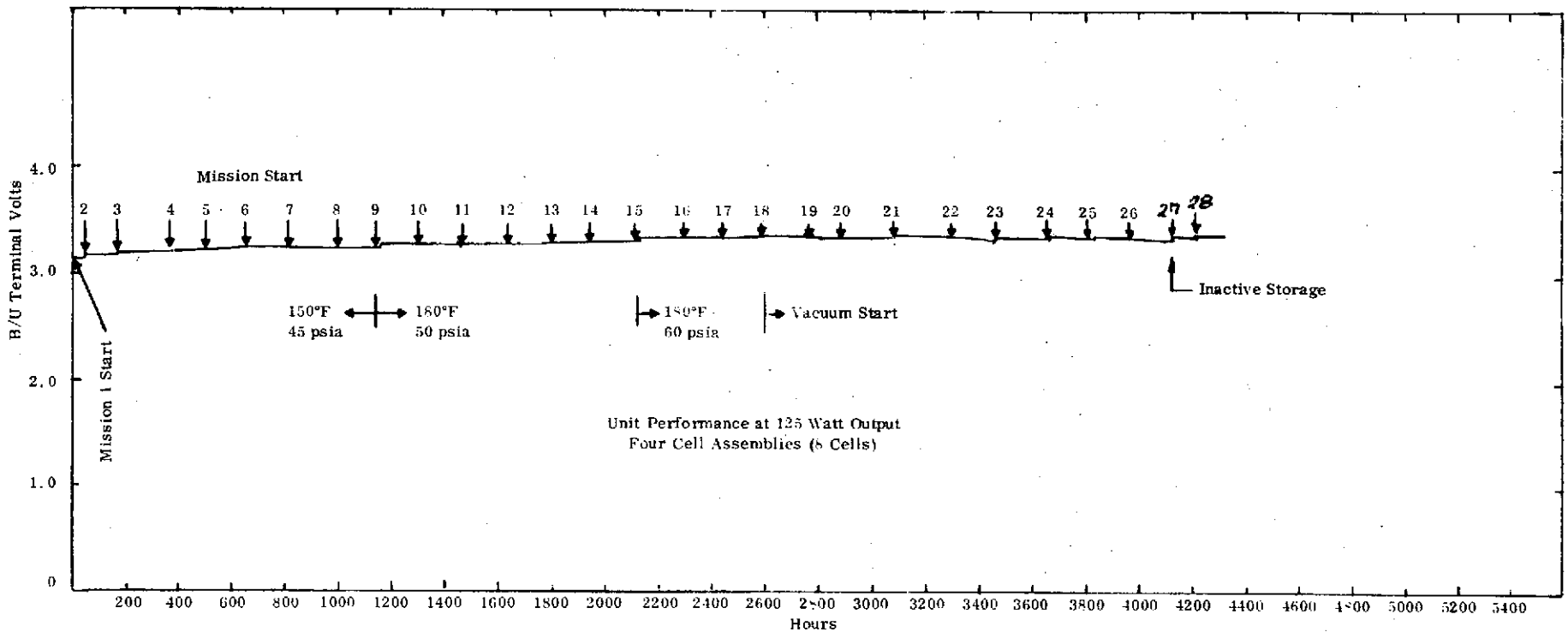


Figure 8. B/U 104 Performance

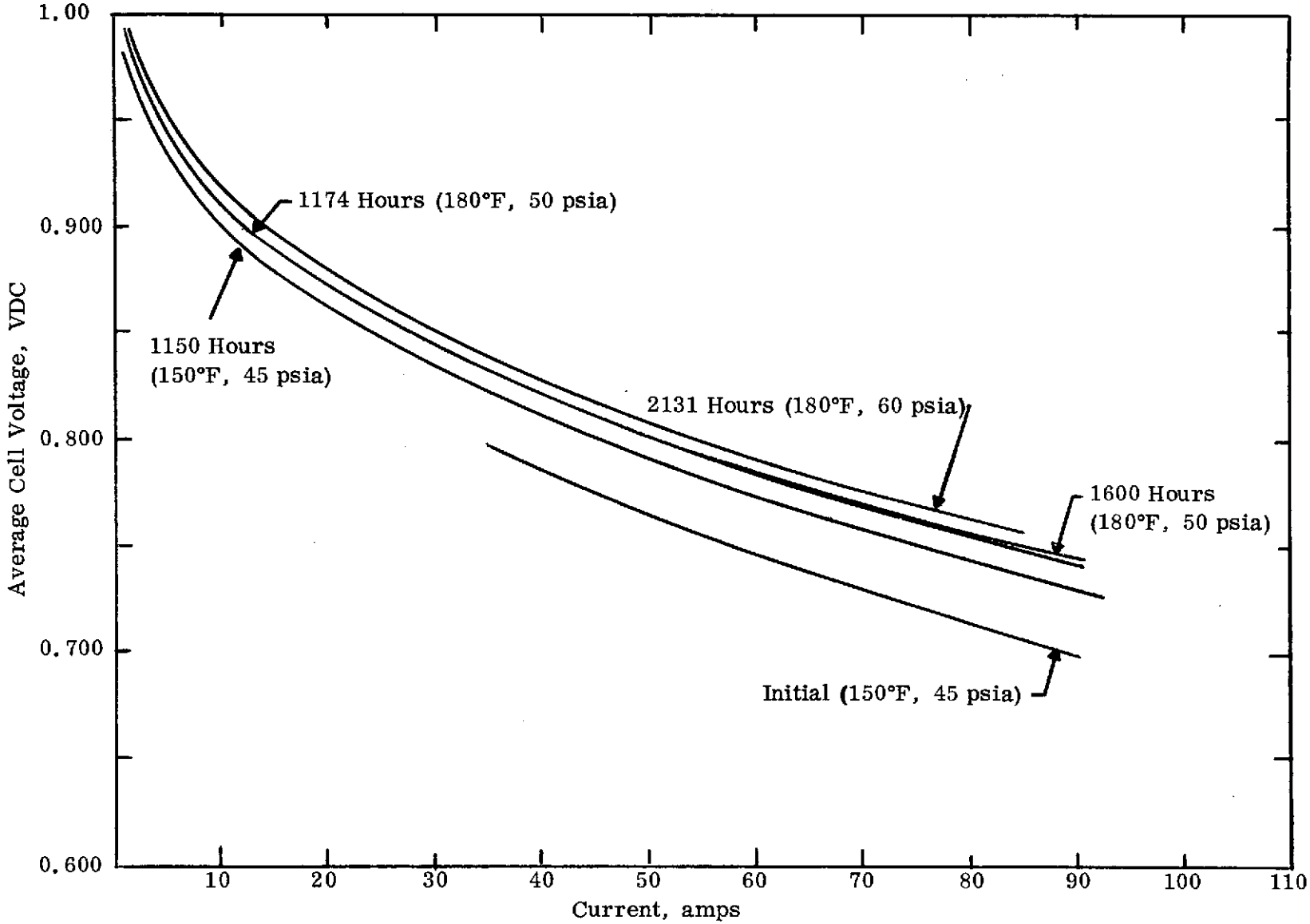


Figure 9. B/U 104 Cell Polarization Data

was equivalent to AF-42. Bonding temperatures and pressures were developed by GE/DECP.

Two of the B/U 105 cells contained additional current collector strips to minimize IR drop within the electrode.

B/U 105 was activated by the deep discharge-electrolysis method. Simulated missions were run at 180°F and 60 psia. At the 157 load hour point, performance levels were somewhat better than previous experience. HF release in the product water was < 1 ppm.

B/U 105 continued on for 520 hours, during which extensive performance mapping was performed. At this time, the following observations were made:

a) Performance improvement was sustained at mission changes through 300 hours. There was no additional improvement after 300 hours.

b) Cells bonded with FM-1000 showed no significant difference over cells bonded with AF-42 bond material.

The buildup was removed from test at 520 hours to install a cell with a different cathode configuration. This "raincoat" cathode (Teflon sprayed on the cathode) configuration had previously (1969) showed performance levels 10 to 15 mv higher than the Space Shuttle cells. B/U 105A was reinstalled in the facility with the new cell in Position No. 1.

Figure 10 shows the "raincoat" cell assembly gave increased performance at current densities of less than 25 ASF, but there was a net decrease observed above 25 ASF.

A series of deep discharge/H_2 pump experiments was performed. This was done by placing nitrogen in the oxygen subsystem and driving the cells electrically in the normal fuel cell direction such that the cell voltage is reversed. This "pumps" hydrogen from the anode to cathode side and allows a determination of the anode performance. The normal anode performance observed eliminated suspicions of CO contamination of the anode.

B/U 105 was tested for a total of 969 hours. It was then removed from test in order to install a "gasketed" cell assembly. The "gasketed" cell assembly uses a Teflon frame with a layer of silicone rubber adhesive on either side. This frame is placed between the cooling cartridge and the cell. The assembly is then pressed together with no heat applied.

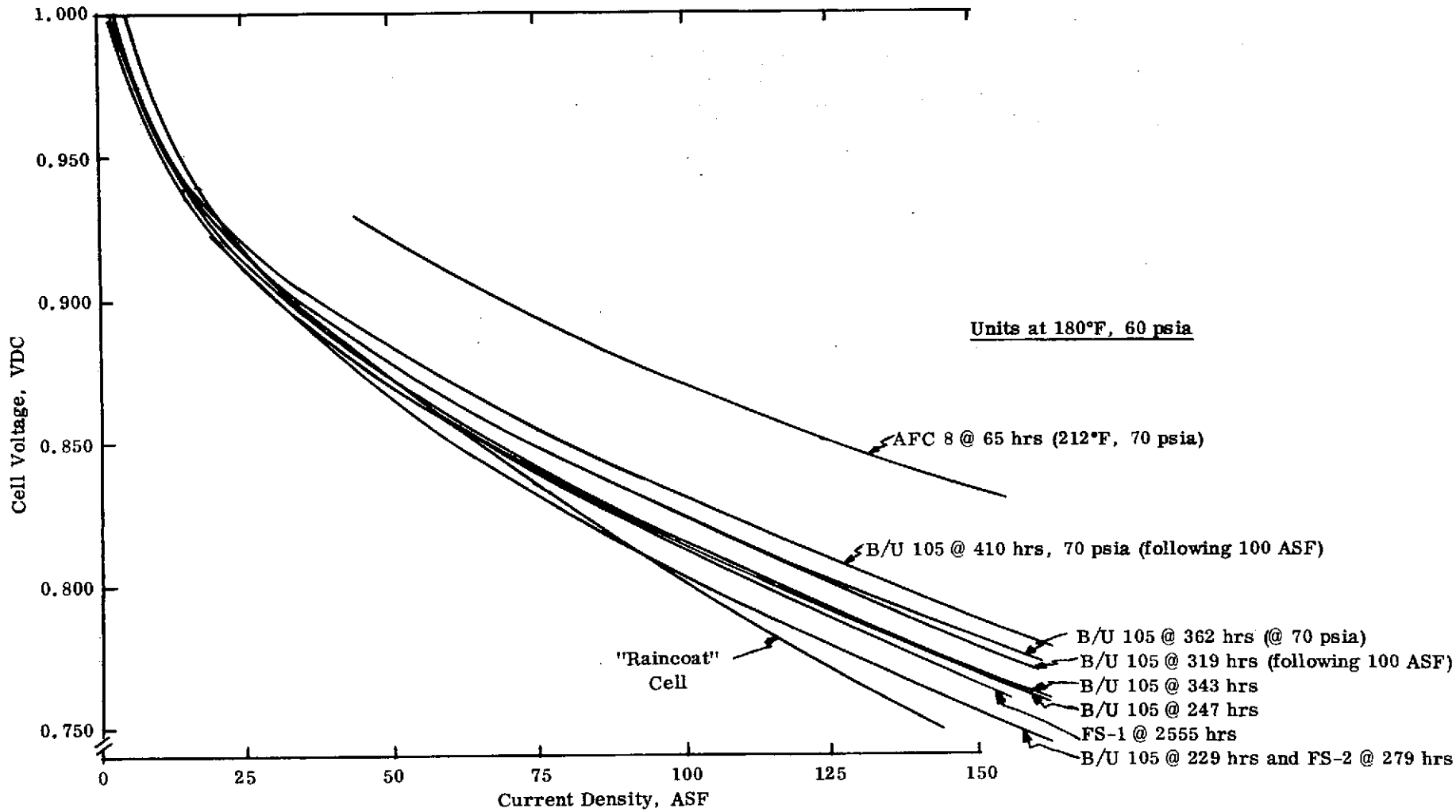


Figure 10. B/U 105 Performance Mapping

The Dacron wicks were replaced with Refrasil glass wicks for evaluation at this teardown. The performance of the "gasketed" cell in Position No. 1 was 10 to 15 mv better than the bonded cells after the new cell had been fully activated. Performance of this unit is shown in Figure 11.

Buildup 105D operated through 1402 hours and 15 simulated missions when it was manually shut down to replace the three bonded cells with Teflon double-sided adhesive cells. Throughout B/U 105D testing (300 hours) the Position No. 1 cell, fabricated using the Teflon double-sided adhesive (TDSA) frames, exhibited the highest performance.

B/U 105E was assembled with all four cells using the Teflon frames. Cell No. 1 of B/U 105D, which had cathode loadings of 8 mg/cm^2, was moved into Position No. 3. The remaining cells had the following cathode loadings:

Cell Position No. 1	2 mg/cm^2
Cell Position No. 2	4 mg/cm^2
Cell Position No. 4	12 mg/cm^2

B/U 105E was activated and operated through 1749 total hours (347 hours as B/U 105E) and 17 simulated missions (two missions as B/U 105E) with performance better than previously experienced on all cells. See Figure 12.

Figure 12 shows very little difference in performance over the 2 - 12 mg/cm^2 cathode loading range. However, it would appear that the 8 mg/cm^2 loading utilized over most of this program is near the optimum loading.

B/U 105E experienced an automatic shutdown in May 1973 after 993 hours of operation. Cell No. 3 of this configuration had been operated for 299 hours in a previous buildup for a total of 1292 hours. The performance of these cells continued to be 20 to 30 mv higher than the bonded cells (Figure 13).

Prior to the shutdown, the HF release in the product water was observed as high as 3 ppm, whereas < 1 ppm is normal for the operational conditions on prehumidified reactants. Reactant humidifier temperatures were rechecked and found to be normal. Laboratory tests on 3 x 3 inch hardware were performed, using the Teflon double-sided adhesive material. These tests showed the normal < 1 ppm HF, indicating that the HF observed was not related to the change in frame and adhesive material.

A teardown of the buildup hardware revealed an oxygen inlet area delamination in all cells and holes in the SPE at the delaminated area. No hydrogen inlet delamination was noted. Since sub-saturated reactants are the only known cause

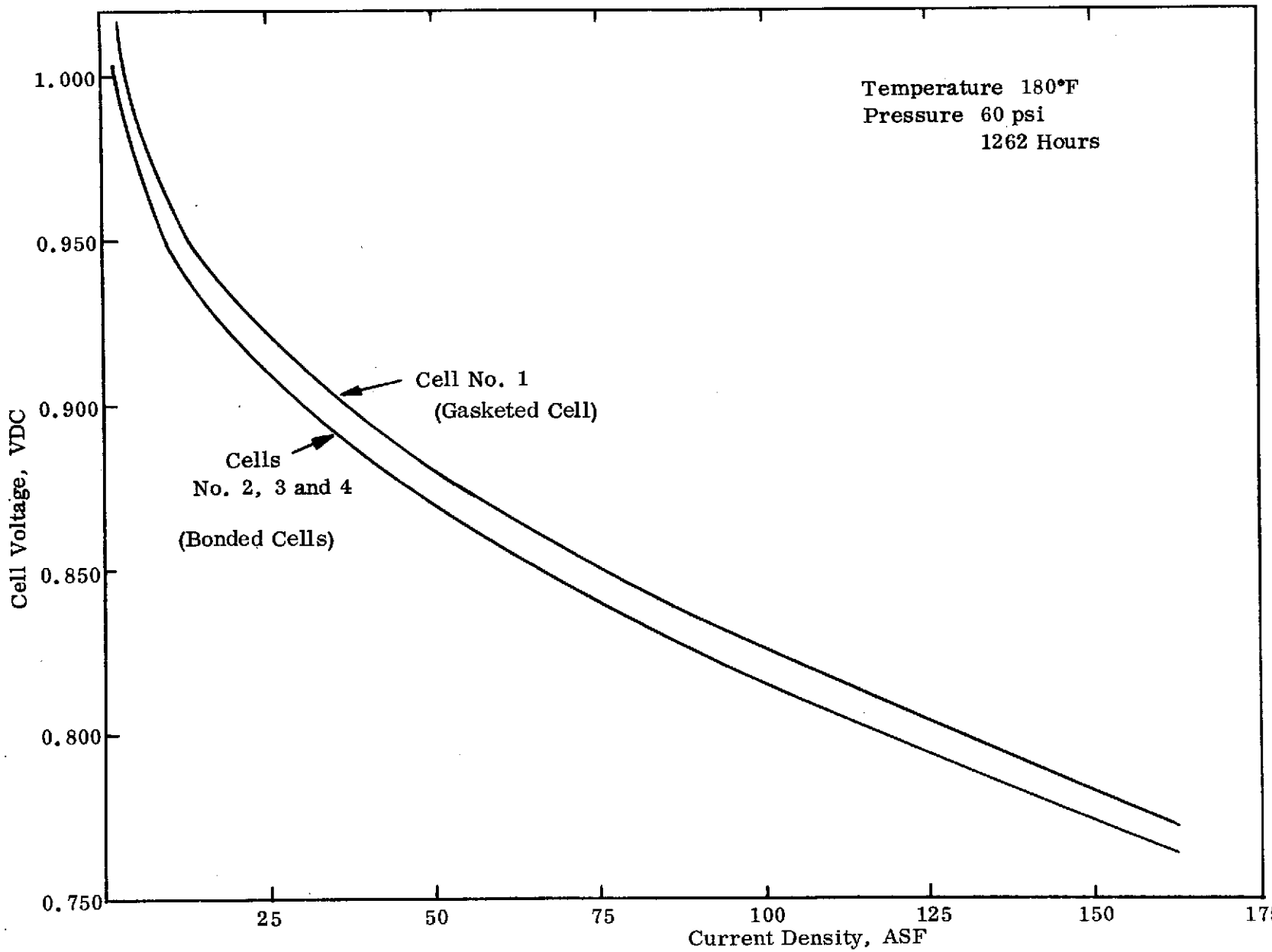


Figure 11. B/U 105D Polarization Data

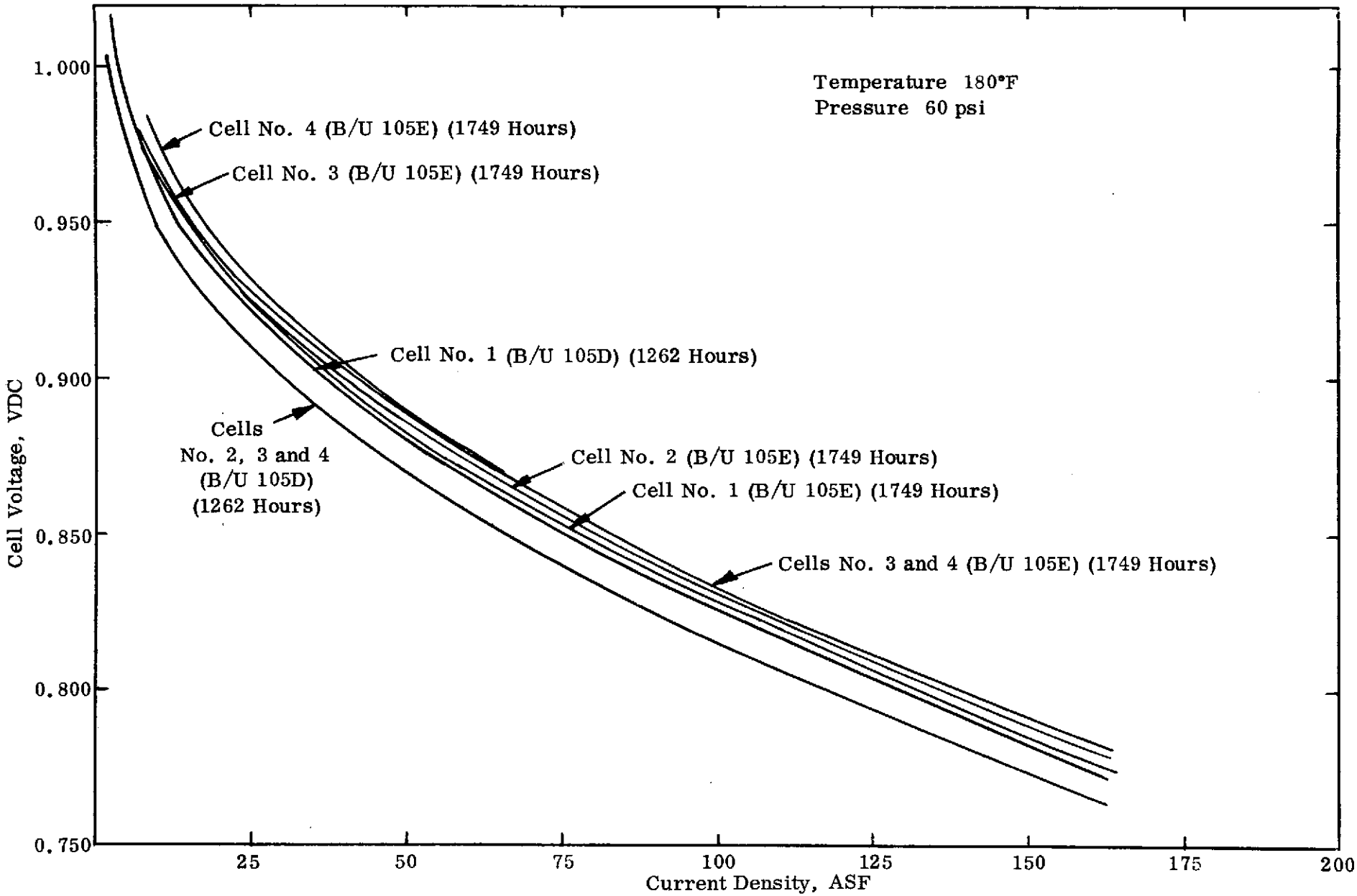


Figure 12. B/U 105 Performance

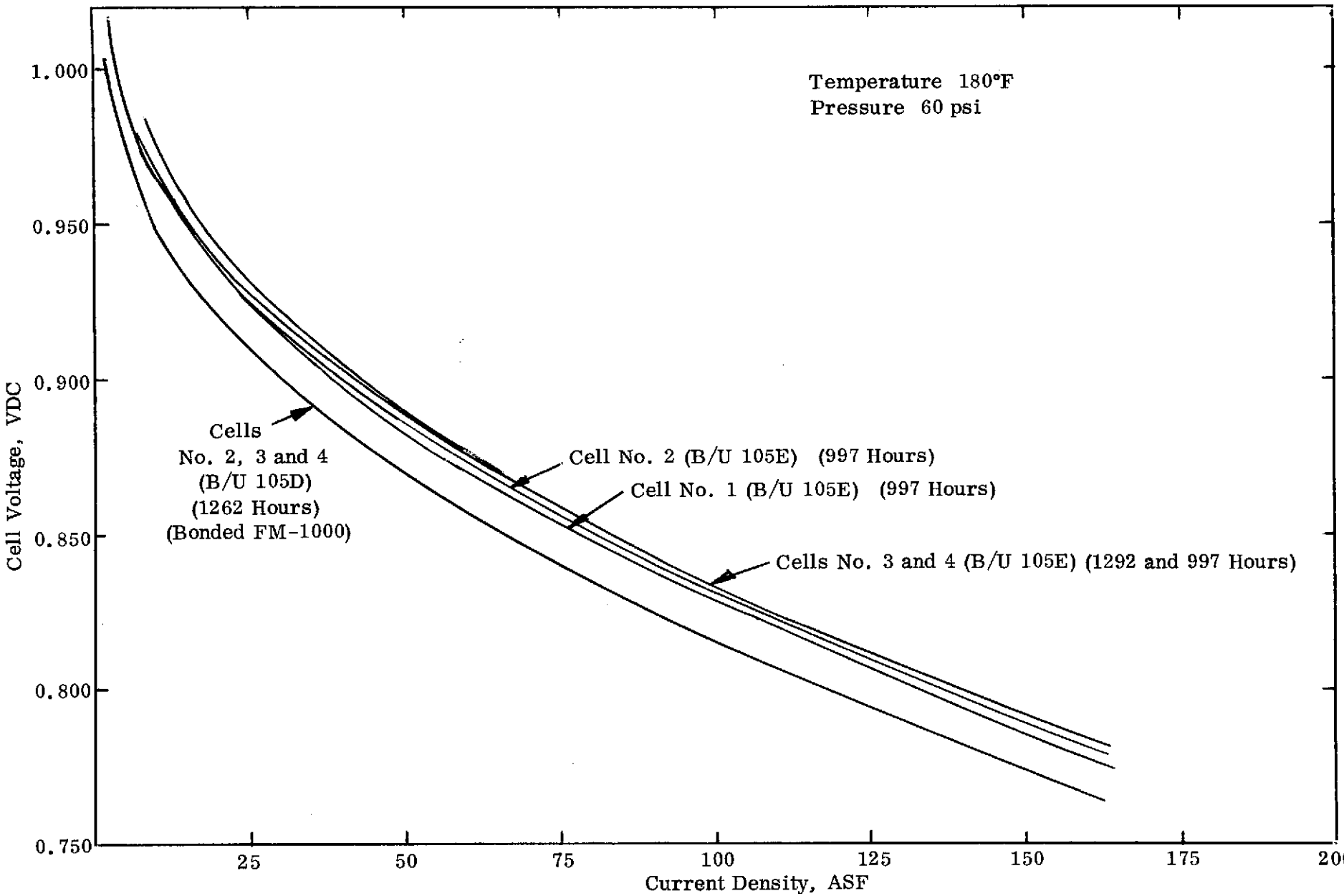


Figure 13. B/U 105 Performance

of membrane delamination, the facility reactant prehumidifiers were thoroughly checked out and found to be producing 100% RH for both reactants. An analysis of the amounts of water collected from the test case revealed that an oxygen relative humidity as low as 50% at 180°F occurred in the test case if the test case water was the result of condensation from the oxygen reactant.

The back panel of the test case was not insulated and could thus be the "condenser" for reducing the water content of the oxygen in the test case. The configuration of this test case allowed the incoming oxygen to flow into the case, and the oxygen entered the stack from the case through an open hole in the end plate. This type of problem does not exist in the full stack configuration since the end plate integrated humidifier pipes the oxygen directly from the humidifier section into the stack.

The teardown of B/U 105E revealed that the Teflon double-sided adhesive peel strength was "like new".

It is concluded from B/U 105E testing that the Teflon frame with double-sided silicone adhesive between the SPE and the coolant cartridge is superior to the earlier AF-42/FM-1000 adhesive/polysulfone frame/ high temperature bond system.

4.1.6 B/U 106

Buildup No. 106 was a small stack of four cell assemblies designed, fabricated and tested with General Electric Company development funding. The design of B/U 106 included increased cell active area (from 0.7 to 0.82 ft^2), lower weight coolant cartridges and the Teflon frame with silicone adhesive as a replacement for the polysulfone frames and AF-42 bonding.

B/U 106 was activated in April 1973, utilizing a facility which was previously operated at 150°F for the life test of B/U 101. Electrical performance was found to be as predicted from B/U 105E performance (see Figure 14). The buildup operated for 576 hours, at which time a low voltage shutdown occurred.

Throughout testing, B/U 106 electrical performance was invariant. However, like B/U 105E, HF release in the product water was observed as high as 5 ppm during the testing. Facility prehumidifiers appeared to have the correct heater power settings, but a unit teardown revealed the tell-tale delaminations and failure point at the hydrogen inlet area and slight delamination at the oxygen inlet area.

Simulated runs of the facility reactant humidifiers, which were of a different design than those utilized for B/U 105, showed that both reactants were maintaining only 50% RH before entering the cells. Figure 15 is a schematic of the

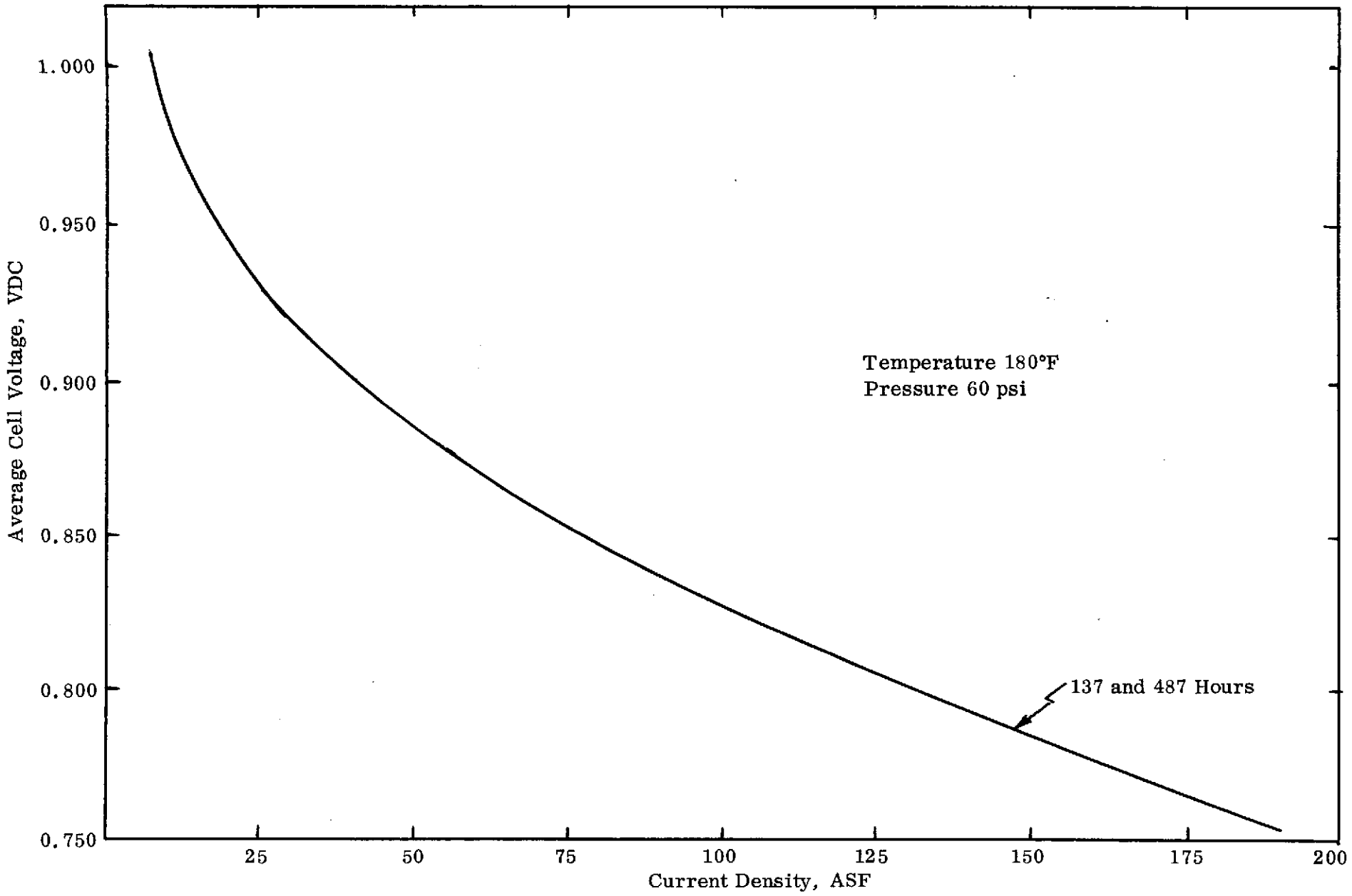
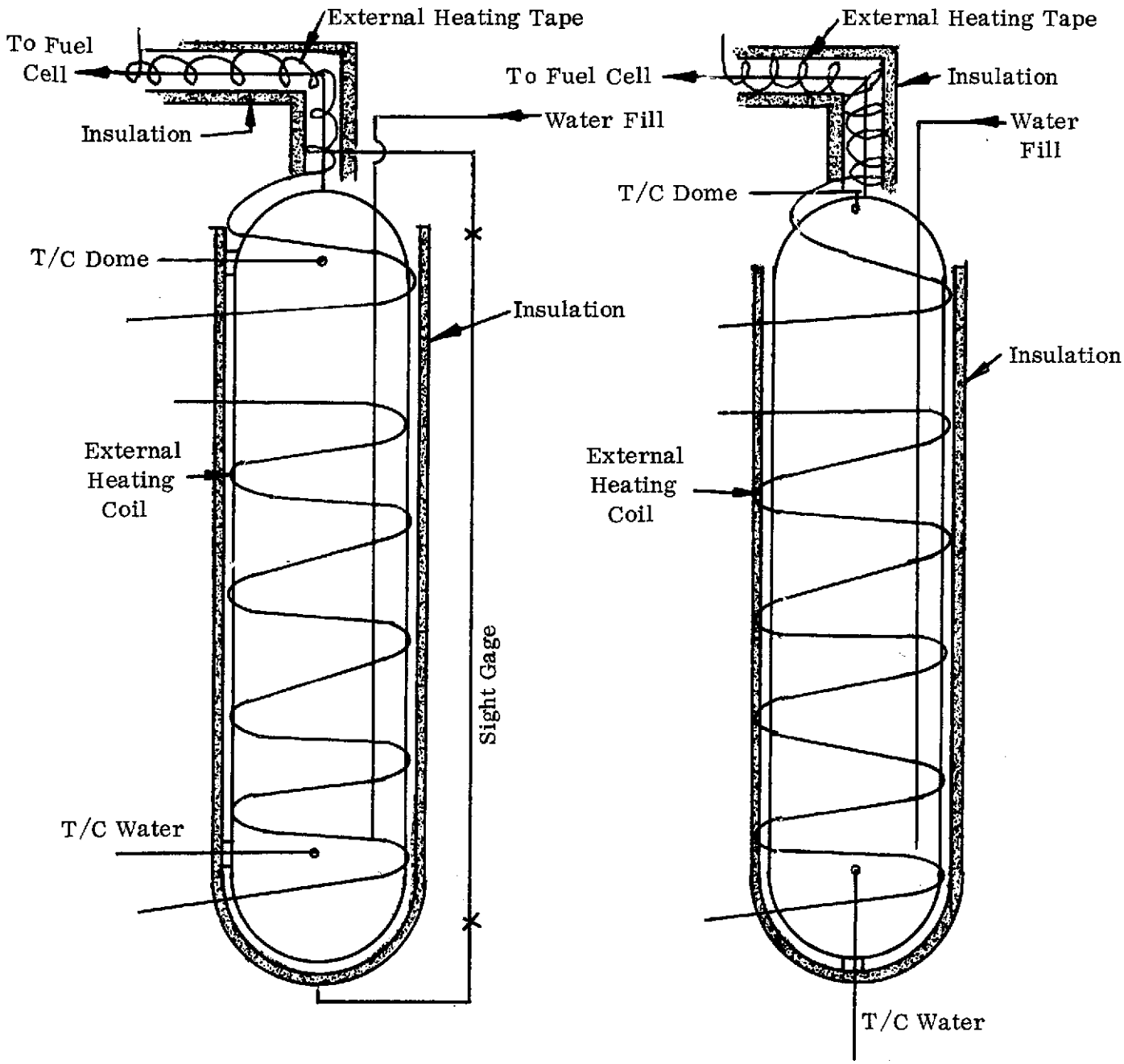


Figure 14. B/U 106 Polarization Data



Stand 6 (B/U 105)	
T/C Water	180°F
T/C Dome	190°F
Dew Point	180°F

Stand 8 (B/U 106)	
150°F	180°F
190°F	190°F
150°F	150°F

Figure 15. Test Facility Prehumidifiers

two prehumidifier facilities used on B/U 105 and 106. Note the difference in location of the thermocouple measuring dome temperature. Apparently, the temperature of the dome was controlling the delivered humidity such that the increase in water temperature did not result in an increase in the dew point of the reactants.

The teardown observations of B/U 106 indicated that the bond strength of the double-sided silicone adhesive on the Teflon frame was "like new". The coolant cartridges also were "like new" and were reused in the buildup of B/U 106A.

B/U 106A was fabricated utilizing B/U 106 hardware and replacing cell assemblies. The facility humidifiers were reconfigured to give 100% RH over all operational temperatures, pressures and flows.

B/U 106A has accumulated over 2200 hours and 19 simulated missions as of this writing.

Performance has been stable and at a level comparable with other buildups using the Teflon frame. See Figure 16. HF release in the product water remained at < 1 ppm.

4.2 Full Stack Testing

4.2.1 Full Stack FS-1

The first full stack (2.5 KW) consisted of 38 cell assemblies fabricated to the same configuration as the cells in B/U 101. Two views of the stack assembly are shown in Figure 17. Instrumentation was added to the stack in order to monitor the voltage of each cell, manifold pressures and internal temperatures.

The stack was assembled into a development test case and installed in the test facilities on 1/7/72, as shown in Figure 18. The ancillary components designed and procured for the prototype EM-1 module were used in the test equipment setup as shown on the schematic drawings of Figure 19. The reactants were prehumidified in saturators external to the stack assembly, in the same manner as used in the test equipment for operating B/U 101 and B/U 104.

The first objective was to operate the stack for 2000 hours under the same conditions as used for the small stack B/U 101, in order to demonstrate a capability of scaleup from a 4-cell assembly stack to a 38-cell assembly stack. The cell performance and the quality of the product water did indeed demonstrate the same characteristics.

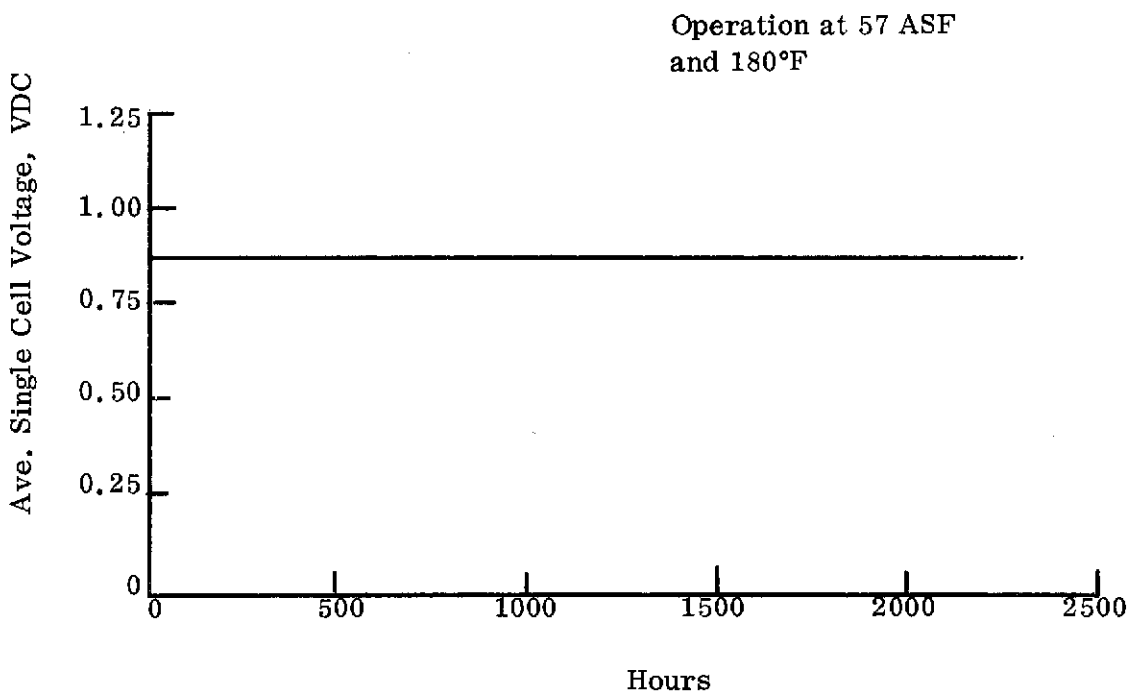
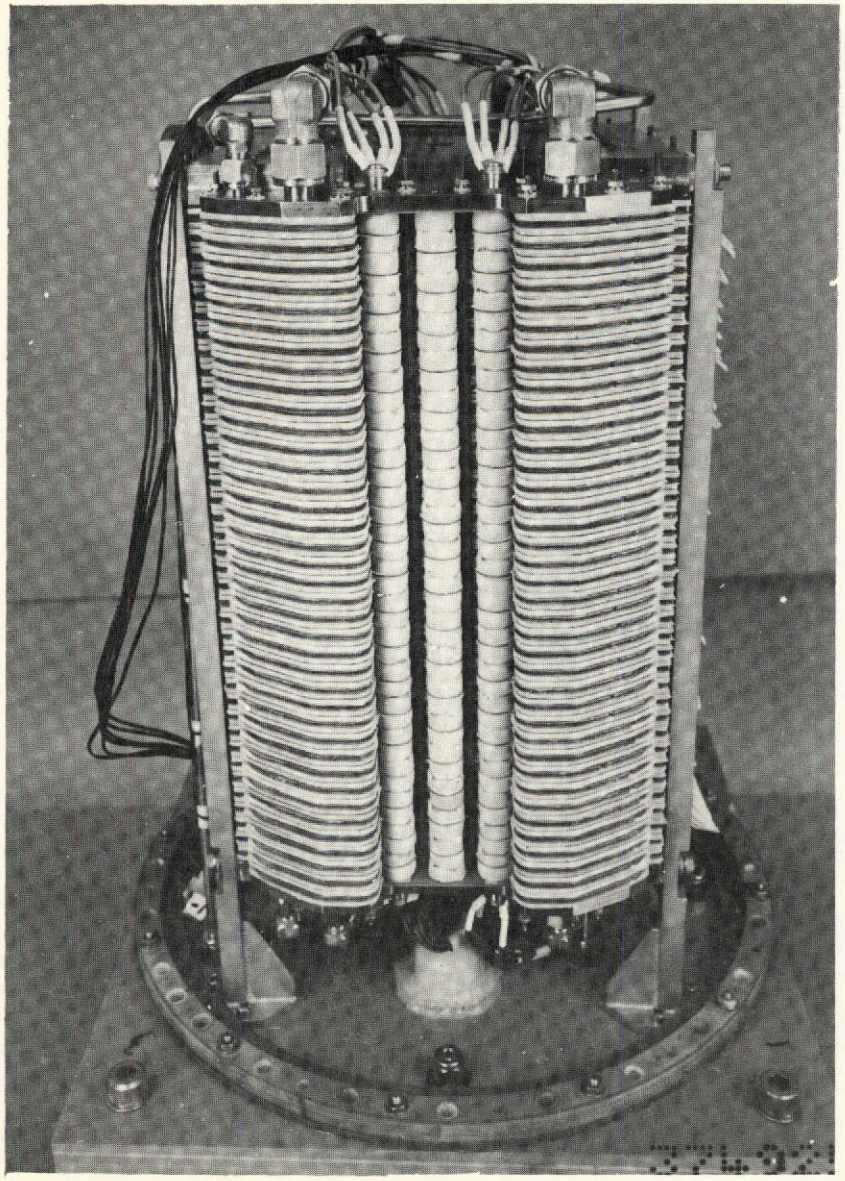
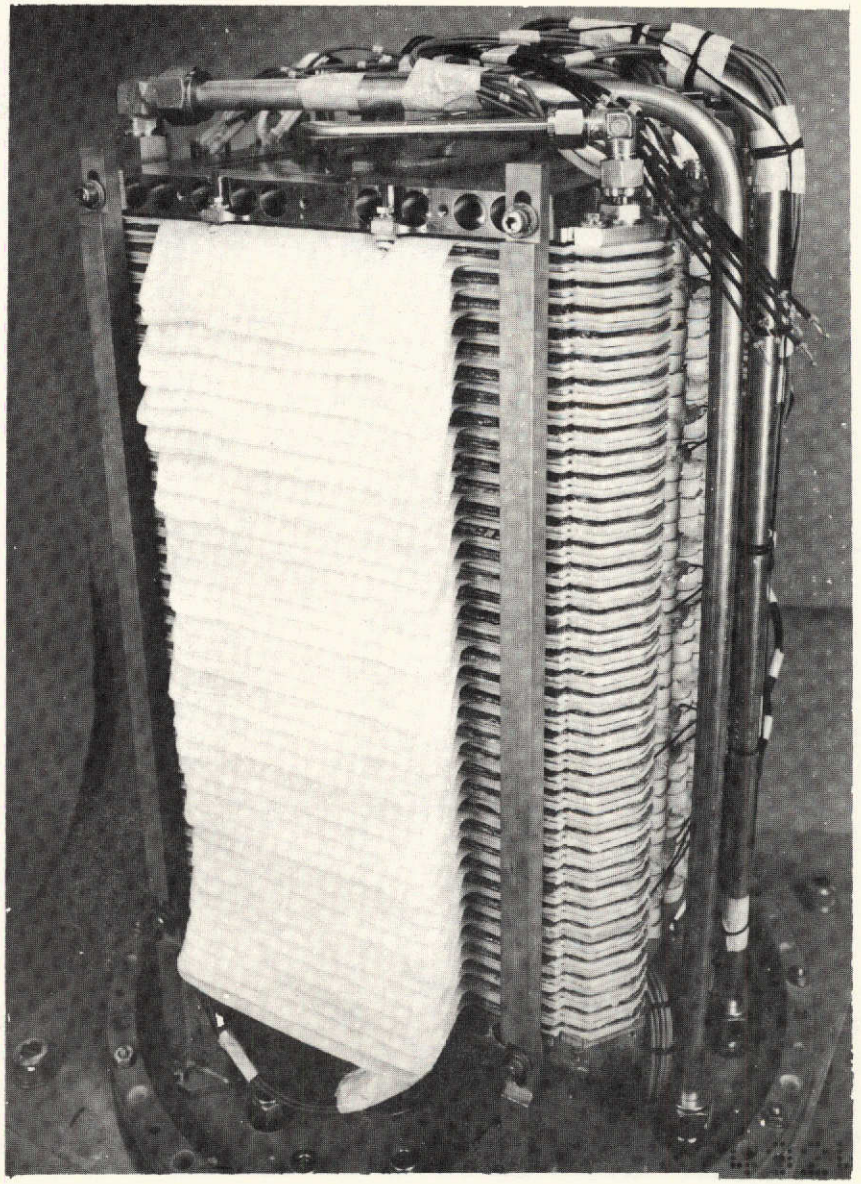


Figure 16. B/U 106A Test Performance



Coolant and Oxygen Inlet Side



Water Removal Wicks

Figure 17. 38 -Cell Stack FS-1

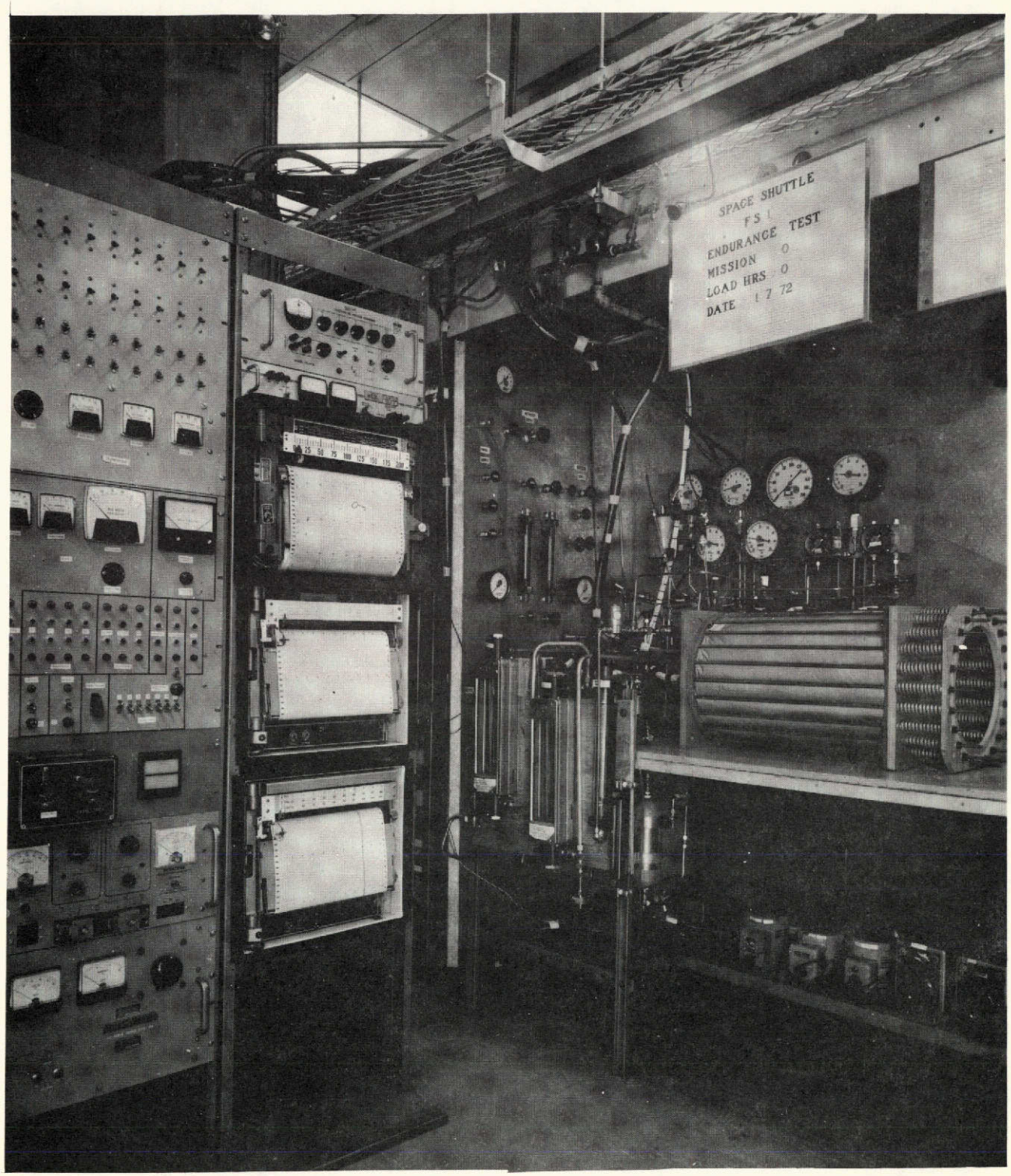


Figure 18. Fuel Cell Stack FS-1 (38 Cell Assemblies) Installed in Test Facility

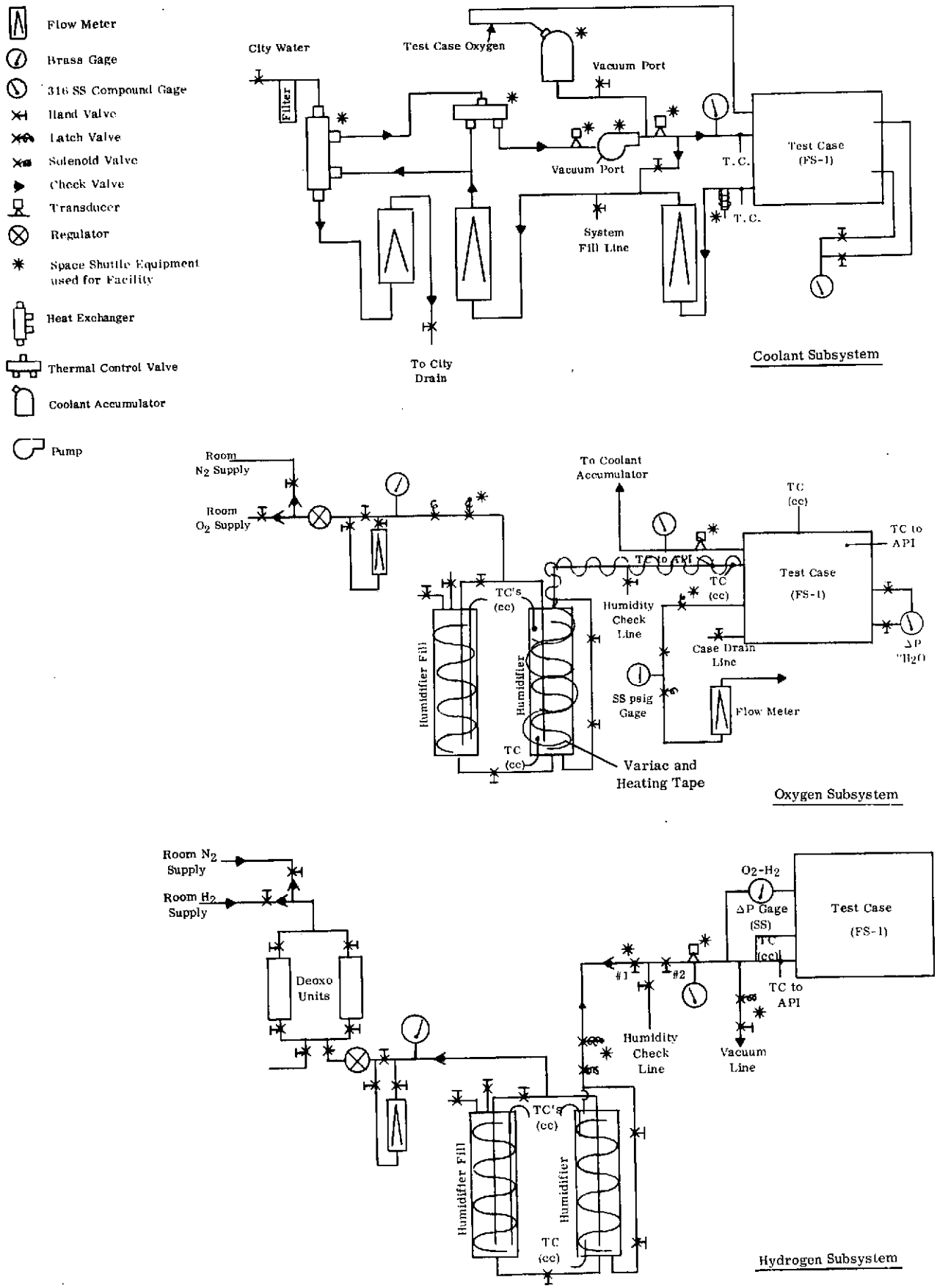


Figure 19. FS-1 Fluid Subsystems

The next objective for the FS-1 full stack was to increase the operating temperature to 180°F and the operating pressure to 60 psia, in order to establish the cell performance capabilities at these conditions. The stack was operated for 750 hours at these conditions with a stable performance improvement of 25 to 35 millivolts, which was again consistent with the data from B/U 101. The HF level in the product water increased to 15 ppm. This was similar to the HF level encountered in the testing of the unplatinized SPE in B/U 103 at 180°F. Since the platinized SPE's in B/U 104 were demonstrating < 1 ppm of HF at 180°F operation at this time, it was decided to lower the operating temperature of FS-1 to 150°F, in order to demonstrate the life correlation between the small stack (B/U 101) and the full stack (FS-1).

The operation of the FS-1 stack was continued for a total of 5000 hours, during which 32 simulated Space Shuttle mission profiles were performed. The performance and life test results shown in Figures 20 and 21 indicate an excellent correlation between the operation of a small stack and a full stack. This data provided a base of confidence for the evaluation of cell assembly configurations at the cost effective level of small stacks.

At approximately the 3500 hour-point in the life test, the FS-1 stack was stored in the oxygen/oxygen mode for five weeks. The performance of the stack after reactivation was identical to that preceding the storage period. Additional performance evaluations in the remaining operating time were made to determine the effects of increased reactant pressure. The results are shown in Figure 21.

During the unit shutdown at the end of Mission 29 (4415 hours), an increase in the oxygen-to-hydrogen leakage/diffusion rate was noted. Six of the 38 cells were removed for laboratory analysis. There was no delamination or chemical degradation of the SPE. However, two cells had small holes in the SPE at the hydrogen inlet area. It was concluded from the analysis that the holes resulted from the technique of drawing gas samples during the oxygen takeover shutdown procedure, resulting in localized hot spots at the inlet areas.

The stack was reassembled with the remaining 32 cell assemblies and returned to life test to complete the 5000 hours of operation. The stack was then removed from the test area and stored in the oxygen/oxygen mode.

The development testing of Full Stack FS-1 is described in GE/DECP Report SPR-111, dated 9 July 1973.

4.2.2 Full Stack FS-2

Full Stack FS-2 (2.5 KW) was fabricated with 34 cell assemblies to the cell configuration previously evaluated in B/U 104, and incorporating an integrated prehumidifier built into the stack end plates. The key distinguishing characteristics

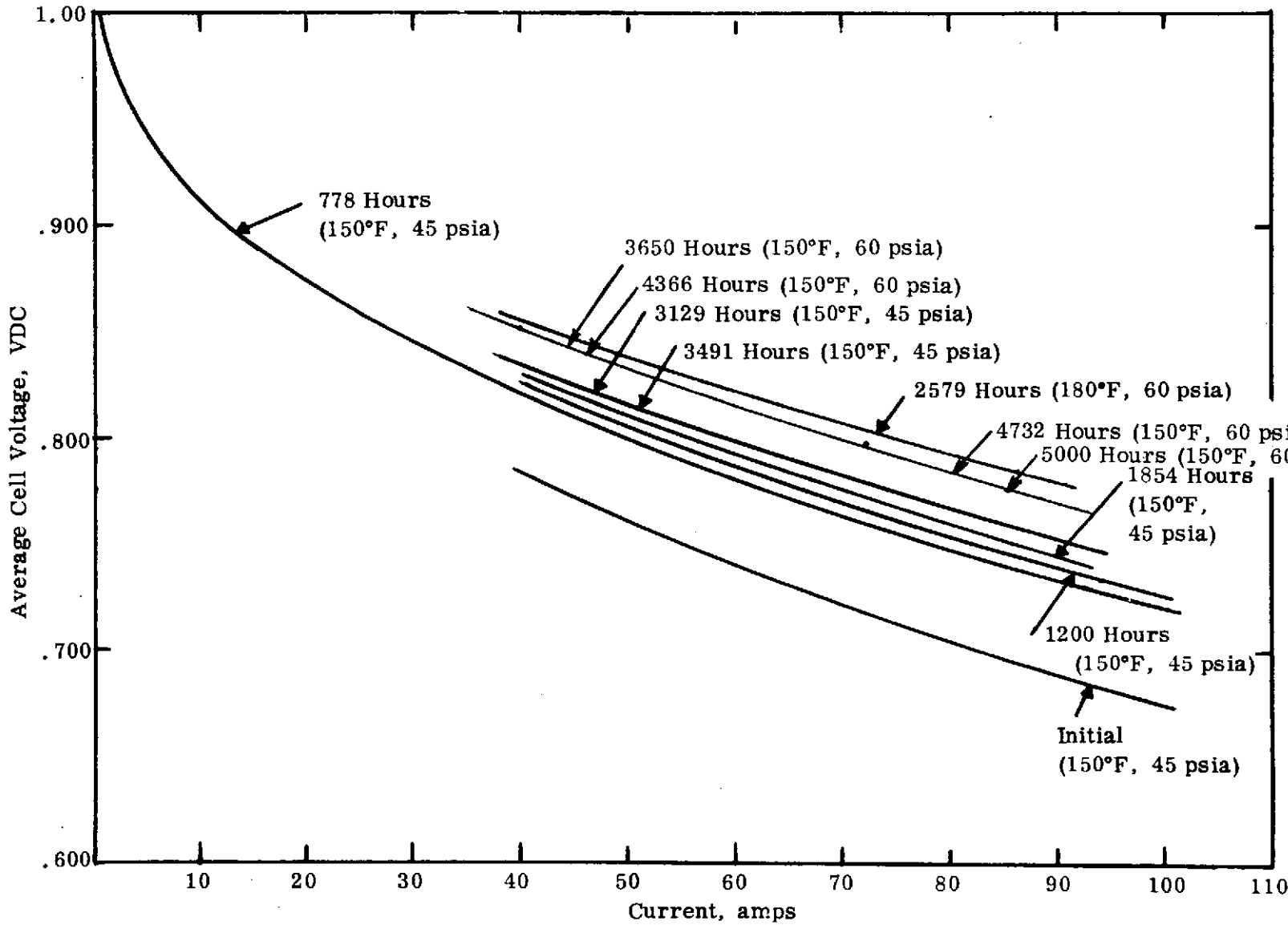


Figure 20. Full Stack FS-1 Performance

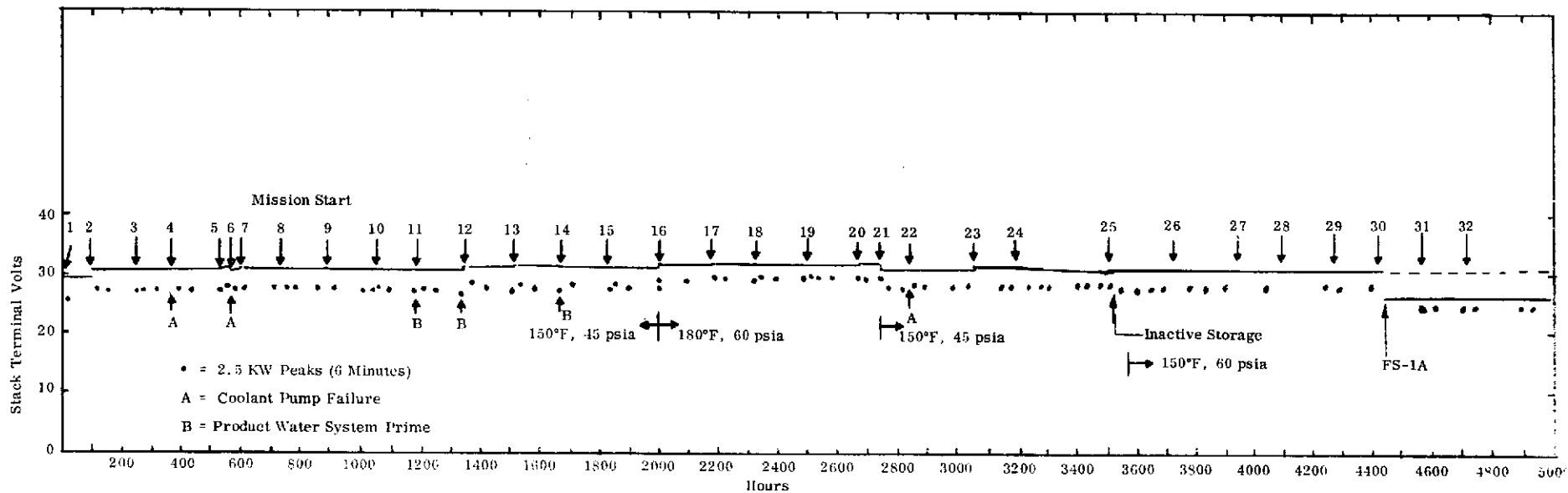


Figure 21. Full Stack (FS-1) 38 Cell Assemblies (76 Cells) Performance at 1.25 KW (FS-1A) 32 Cell Assemblies (64 Cells) from 4415 Hours on

of this configuration are as follows:

(a) Platinized SPE

(b) Anode catalyst loading of 30 mg/cm^2

(c) Cathode catalyst loading of 8 mg/cm^2

(d) Single H_2 inlet channel per cell assembly on same side as coolant inlet and water wick outlet.

(e) The coolant cartridge mid-frame was a single 40-mil thick polysulfone rather than two 20-mil laminates.

This stack was initially paired with Full Stack FS-3 of the same configuration and assembled into a development test case for the preliminary evaluation of the Engineering Model. When the FS-3 stack experienced a failure during the acceptance test procedures, the FS-2 stack was operated separately to evaluate the performance of the integrated end plate prehumidifier. Performance tests were run for two mission cycles (328 hours) at 180°F and 60 psia. Polarization performance was as predicted. (See Figures 22 and 23.) The HF level in the product water remained at < 1 ppm, indicating adequate reactant saturation from the prehumidifiers. The FS-2 stack was then deactivated and stored in the normal oxygen/oxygen storage mode to await reassembly into EM-1, along with a new 34-cell stack (FS-4).

In the course of acceptance testing EM-1 with stacks FS-2 and FS-4, an automatic test shutdown was experienced when one cell assembly in FS-2 was suddenly driven negative. Subsequent teardown analysis revealed that the cell assembly had a blocked hydrogen inlet tube. Teardown analysis revealed the one cell assembly was leaking due to a crack in the polysulfone frame between the SPE and the coolant cartridge. This is the same condition which had caused the initial failure of stack FS-3 in EM-1. Both stacks FS-2 and FS-3 were fabricated in the same production run and to the same configuration. The details of failure analysis for stacks FS-3 and FS-2 are given in reports SPR-098, dated 19 December 1972, and SPR-101, dated 7 March 1973, respectively.

The six cells removed from FS-2 for analysis were replaced with undamaged cell assemblies from FS-3. The entire assembled stack was coated with RTV-118 to seal any remaining frame cracks. The stack was returned to test in a development container for additional performance testing, transient response tests and short circuit tests. This stack accumulated a total of 852 hours of operation at 180°F and 60 psia. It was then shutdown in the oxygen/oxygen storage mode.

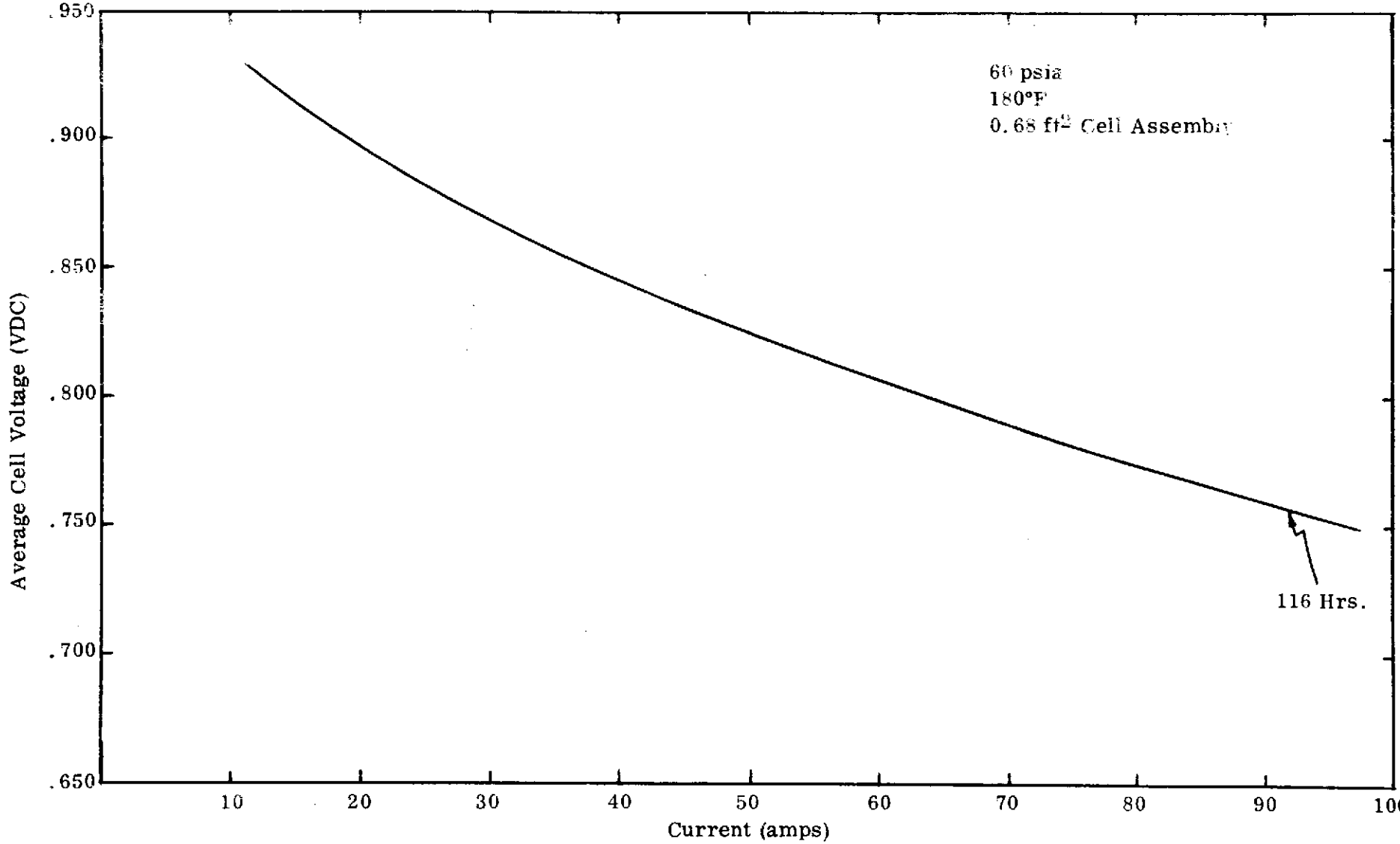


Figure 22. Full Stack FS-2 (34 Cell Assys/68 Cells) Polarization Data

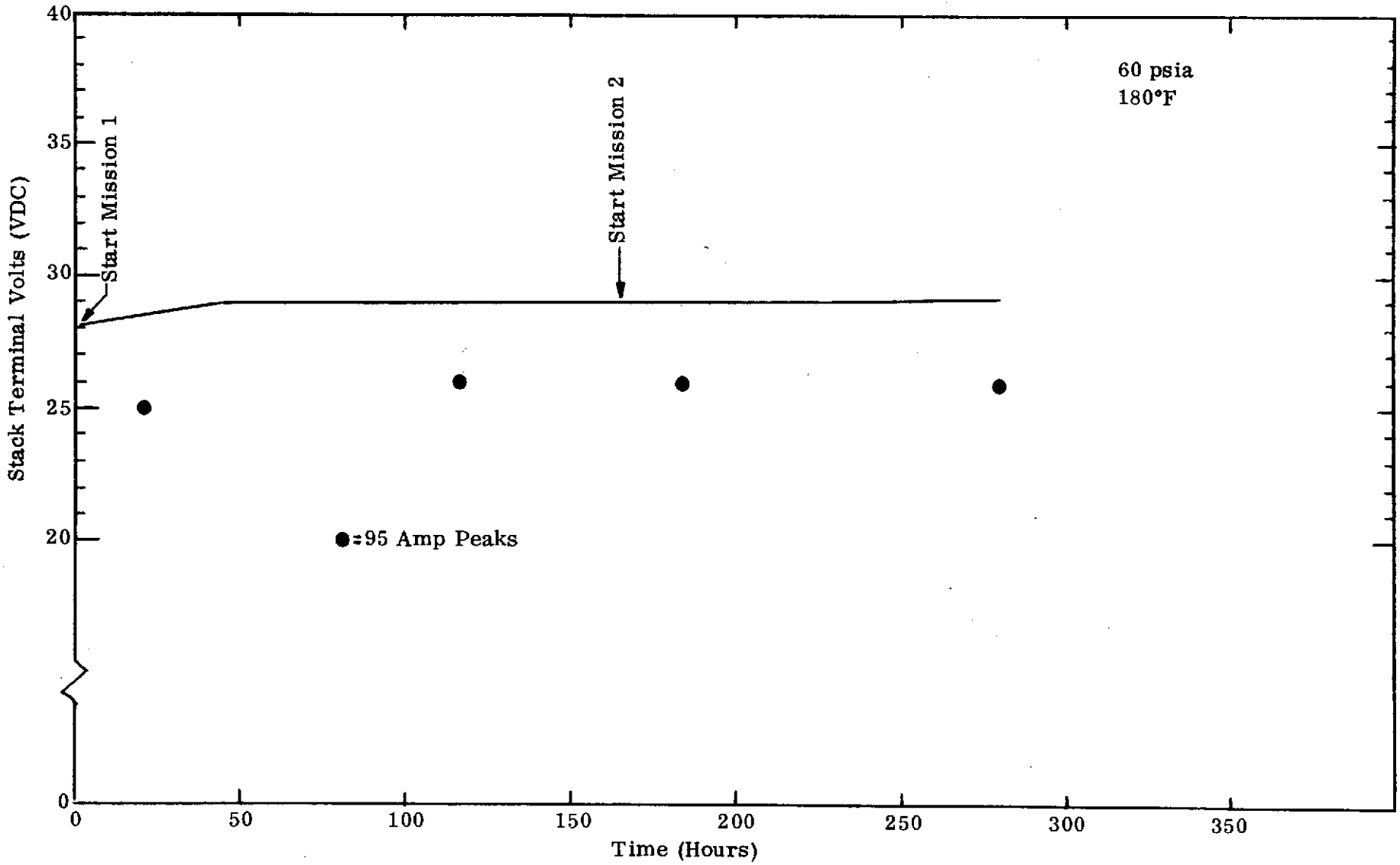


Figure 23. Full Stack FS-2 (34 Cell Assys/68 Cells) Performance at 34 Amps

4.3 Engineering Model Testing

The initial assembly of the 5 KW Engineering Model (EM-1) consisted of two 34-cell stacks (FS-2 and FS-3) assembled into a development test case. The cell assemblies of these two stacks were of the same configuration as small stack B/U 104, i.e., the solid polymer electrolyte was platinized. The stack assemblies had a single hydrogen inlet manifold on the coolant inlet side of the cell assemblies, and the integral reactant prehumidifiers were built into the stack end plates.

The EM-1 unit was set up in the test facilities and the acceptance tests initiated in accordance with Test Plan SPR-084, dated 14 July 1972. Leakage and activation tests indicated excessive O_2-to-H_2 leakage at operating pressures. The leakage was located in Cells 21 and 31 of stack FS-3 and identified as coming through cracks in the 0.020 inch polysulfone spacer frame between the cell and the cooling cartridge. These two cells were replaced.

The unit was returned to test with satisfactory leakage and activation evaluations. During the first 12 hours of performance evaluation of the acceptance test, an automatic shutdown of the test occurred, and gross O_2-to-H_2 leakage was discovered in stack FS-3. Subsequent analysis of the failure concluded that Cell 20 developed a cross-cell leak during operation as a result of damage incurred during the initial activation when the cell was adjacent to the leak found in Cell 21. Inspection of the remaining cells in the stack revealed that Cell 30 showed signs of localized heat damage from being adjacent to the other original leakage point at Cell 31. Although Cell 30 showed signs of damage, it did not leak. It was concluded that both Cells 20 and 30 were similarly damaged by their proximity to the leaking cells. This condition was not detected in the rebuild because they did not leak at that point, but the subsequent operational stresses caused Cell 20 to fail.

As a result of this failure, improvements were made in the cell assembly bonding fixtures to reduce the thermal expansion stresses on the polysulfone frames. A high pressure differential (60 psid) cross-cell leak test was also instituted in the assembly area for both cell assemblies and stack assemblies. This procedure used dry nitrogen in order to have a more descriminating quality test before subjecting the units to operational tests. A failure analysis report of EM-1 is given in GE/DECP report SPR-098, dated 19 December 1972.

A new stack (FS-4) was fabricated for installation in a flightweight prototype container assembly. This stack incorporated the corrective actions identified above and confirmed with stack FS-2. The ancillary components were integrally mounted to the container. The Engineering Model 5 KW prototype was designated EM-1A. See Figures 24 and 25.

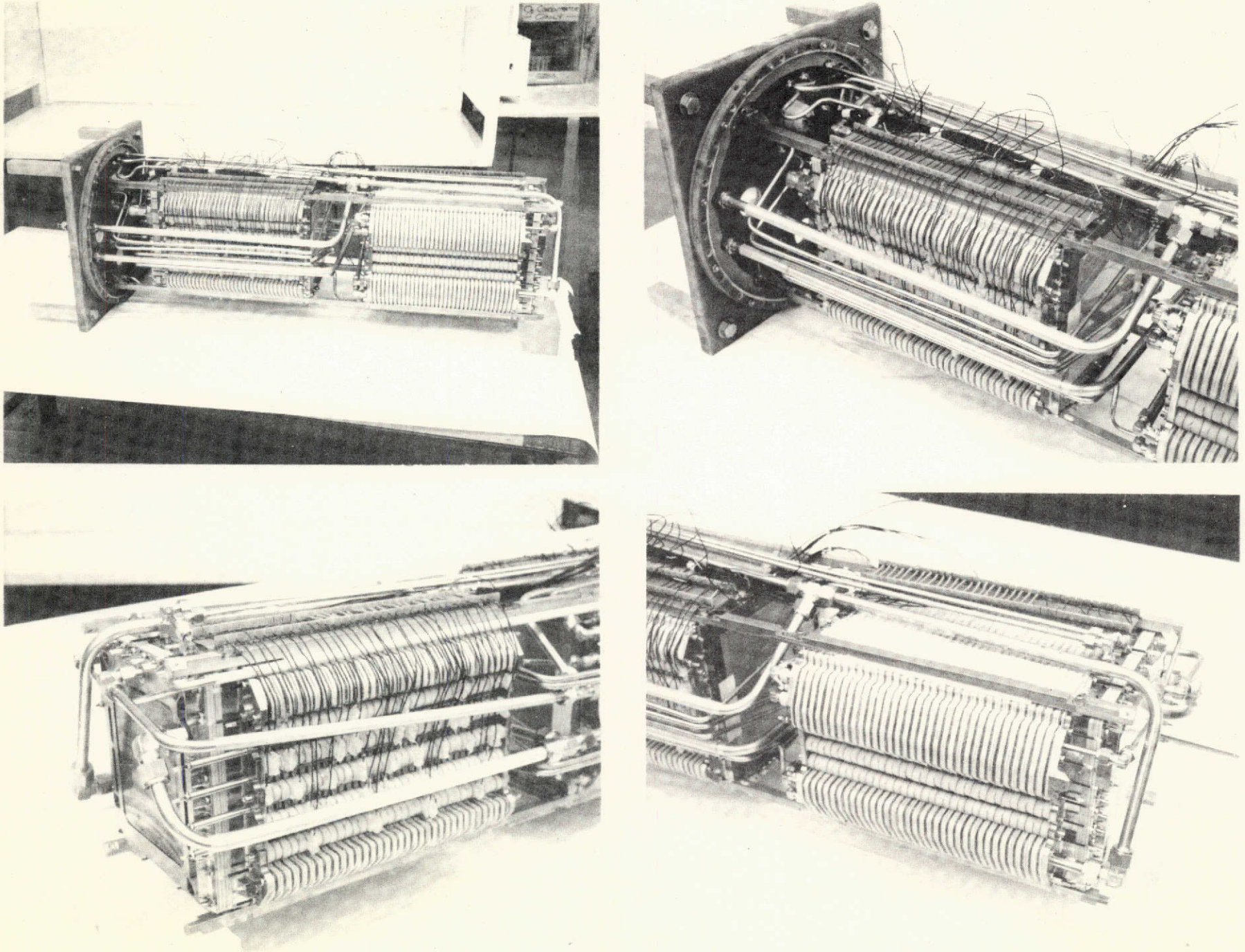


Figure 24. Engineering Model EM-1A Stacks

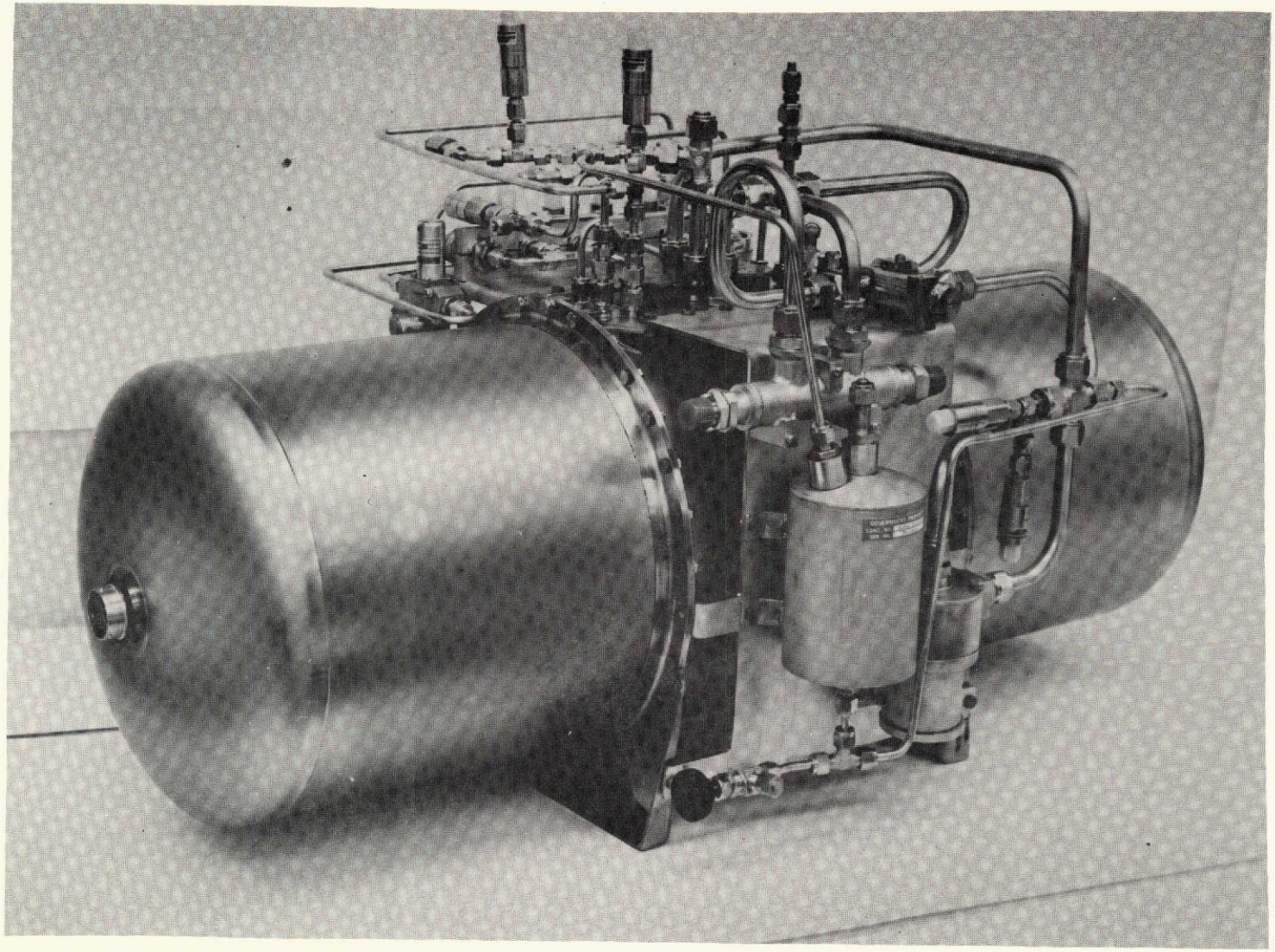


Figure 25. Engineering Model EM-1A with Ancillary Components

During the acceptance testing at a peak load point, Cell 21 of stack FS-2 was driven negative and developed excessive O_2-to-H_2 leakage. This leakage was also subsequently analyzed as the result of a cracked polysulfone frame between the cell and the cooling cartridge. The cell assemblies in stacks FS-2 and FS-3 were fabricated in the same production run and thus equally subject to cracking of the polysulfone frames during fabrication. Cracking of the frames did not always become immediately evident, even in the high pressure testing, since the bonding adhesive flowed to cover the ends of the cracks. As the unsupported bond flash deteriorated in physical properties with time, the cracks could become open passages for the flow of oxygen into the hydrogen compartment of the cell, resulting in the ultimate unpredictable failure.

The failure analysis of EM-1A is given in GE/DECP report SPR-101, dated 7 March 1973.

The module was reassembled as EM-1B with the single stack FS-4 and a dummy block simulating a second stack into the flight prototype container with the integrated ancillary components. After 502 hours and four simulated Space Shuttle missions, Cell 32 became unstable during peak load applications. The cell was removed for examination and found to be partially blocked in the hydrogen inlet tube by excess flash from the bonding adhesive. All cells in the stack were modified to trim back the flash, enlarge the manifold hole in the gasket, and to insert a Teflon washer in the manifold hole to prevent the gasket from bearing close to the manifold.

Close evaluation of the cell assemblies at this time revealed cracks in the polysulfone frames even though no leakage had been evident during operation. The new cell selected to replace Cell 32 also showed signs of cracks, although it had never been assembled into a stack. This observation again confirmed the fact that the polysulfone frame cracking problem occurs during the cell assembly fabrication rather than during stack assembly or operation. The bonding flash was removed from the edges of the cell assemblies, and after stack compression, the edges were coated with RTV-108. During the shutdown at the end of Mission 6 (608 hours), it was evident that excessive leakage was present in the area of Cell 27. It was found that the RTV-108 was too viscous to adequately patch the frame cracks. Consequently, the process was repeated using RTV-118, while drawing a vacuum on the inside of the cell assemblies.

The life testing of the Engineering Model then completed the 2016 hours and 8 more simulated missions with invariant performance (Figure 26). The ancillary components, the pump inverter, and monitoring control unit demonstrated more than 2000 hours of fault-free operation.

The polarization curves (Figure 27) show EM-1B performance in terms of voltage and current. Efficiency and reactant consumption remained unchanged through-

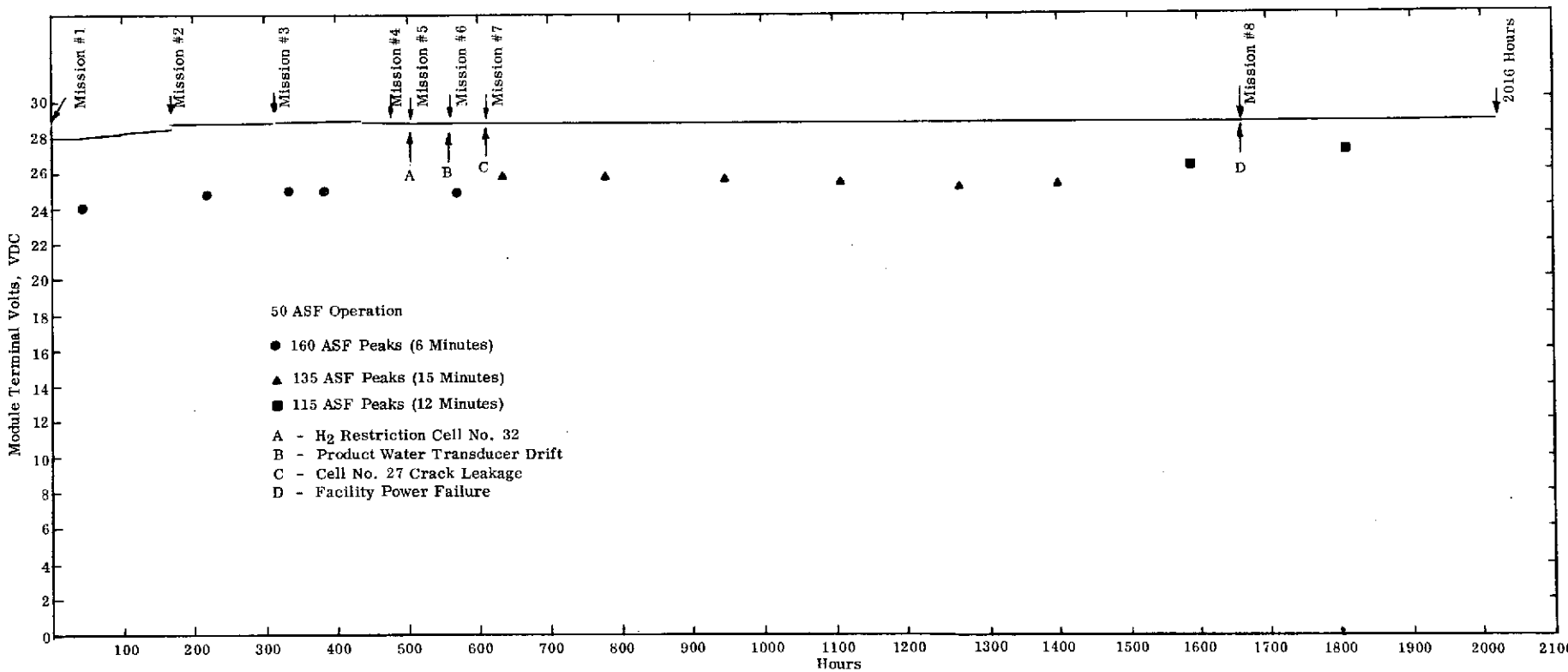


Figure 26. Module EM-1B Operation

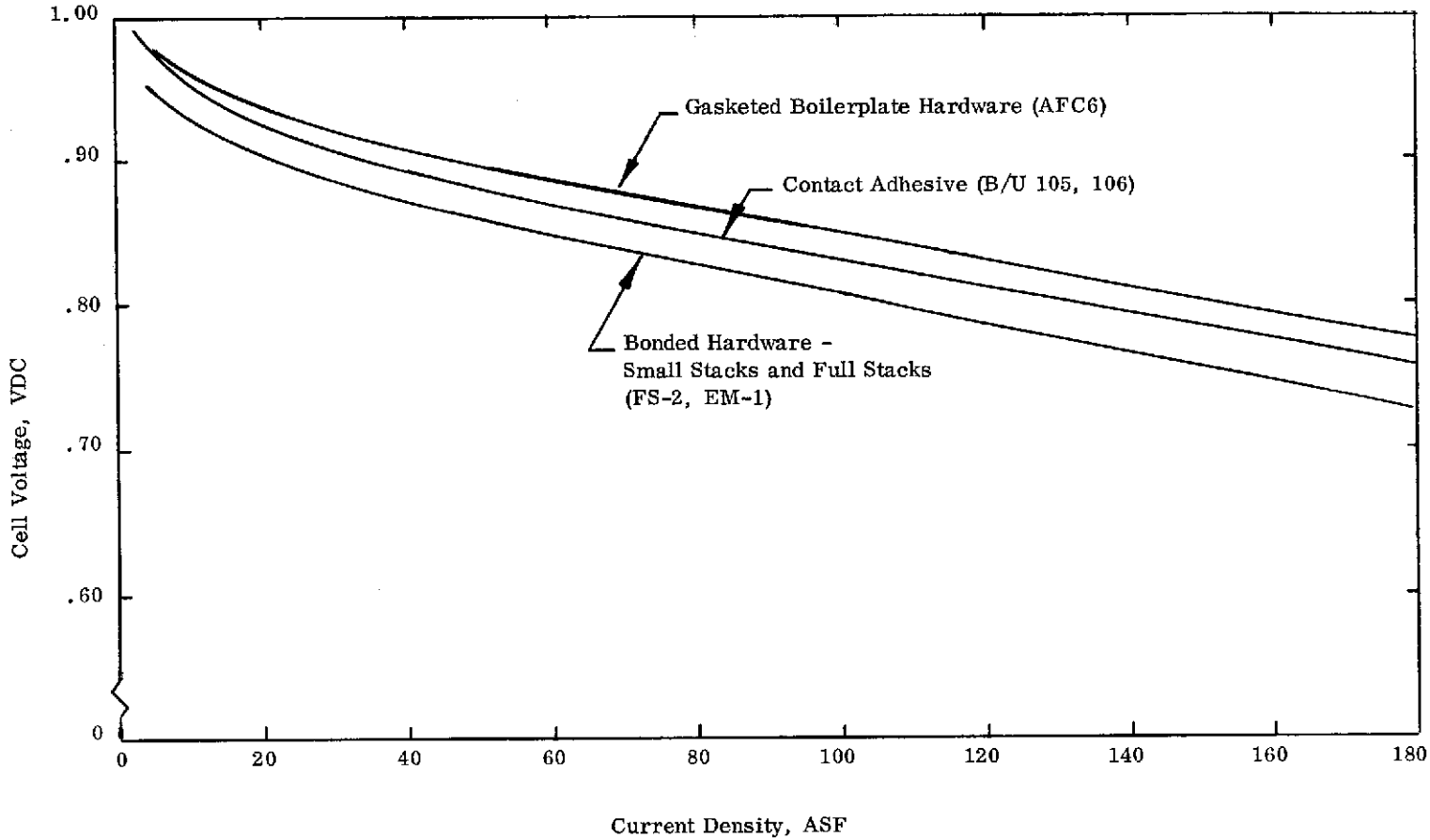


Figure 27. Space Shuttle Hardware Polarization Curves (180°F Operation)

out the 2000 hours of testing, despite the two teardown maintenance cycles performed as previously described. The quality of the product water remained constant, with the fluoride content of the product water remaining less than 1 ppm throughout. Thus, it was substantiated that the integrated prehumidifier built into the end plate successfully performed its function of saturating the reactants before entering the cells. The test results also correlated with the previous experience on B/U 104 with the platinized solid polymer electrolyte, where the fluoride level of the product water while operating at 180°F and 60 psia remained at less than 1 ppm throughout its operating life.

The testing of the Engineering Model is described in GE/DECP Report SPR-112, dated July 20, 1973.

4.4 Component Testing

4.4.1 Fluid Subsystems

The fluid subsystems of the fuel cell module were subjected to life tests at 180°F and operating pressures and flows. The subsystems tested were:

Hydrogen Subsystem
Coolant Subsystem
Prehumidifier, Oxygen and Product Water Subsystem

These components, except for the reactant prehumidifier and the water pressure regulator, previously had successfully undergone more than 2000 hours of testing at 150°F (see SPR-048, dated 25 September 1971).

4.4.1.1 Hydrogen Subsystem

A fuel cell module hydrogen subsystem was tested for 2014 hours over 12 simulated mission cycles to determine failure rates, materials compatibility, and performance characteristics at 180°F operation conditions. All of the subsystem components had previously undergone a successful test for 2016 hours at 150°F, for a total of 4030 hours.

The hydrogen subsystem components tested were:

Hydrogen pressure regulator
H_2 latching valve
H_2 purge valve
Check valve
Transducers (2)
Pressure switch

The components were installed in an oven to achieve the 180°F operating condition. Figures 28 and 29 show the test schematic and the components installed in the oven. Prior to the start of testing, H_2 latching valve S/N 1 exhibited overboard leakage due to a defective O-ring seal. The failure was confirmed by the vendor. The poppet was reworked and a new O-ring installed before starting the endurance test.

The hydrogen pressure regulator had been modified after the 150°F by addition of an inlet filter screen and passivation of the bellows. This was done to prevent possible bellows leakage as had occurred in a water pressure regulator in the 150°F component subsystem testing.

Prior to the start of test, steady state hydrogen flow rates were reduced from 24 to 2.4 liters/min. and time at high flow (56 liters/min.) cut from 15 to 5 minutes in order to save gas without seriously compromising the test objectives.

Testing was terminated after 2014 hours of environmental endurance operation, which included 12 cycles of humidity, temperature and pressure variations which simulated those encountered on Space Shuttle missions.

Performance traces of the hydrogen regulator O_2-to-H_2 differential pressure for Cycles 3 through 12 showed adequate subystem performance at all required levels of pressure, temperature and flow. No detrimental effects were evidenced over the 2014 hours as a result of either cyclic or steady state environmental conditions. All components demonstrated satisfactory performance characteristics, adequate material compatibility and no failures.

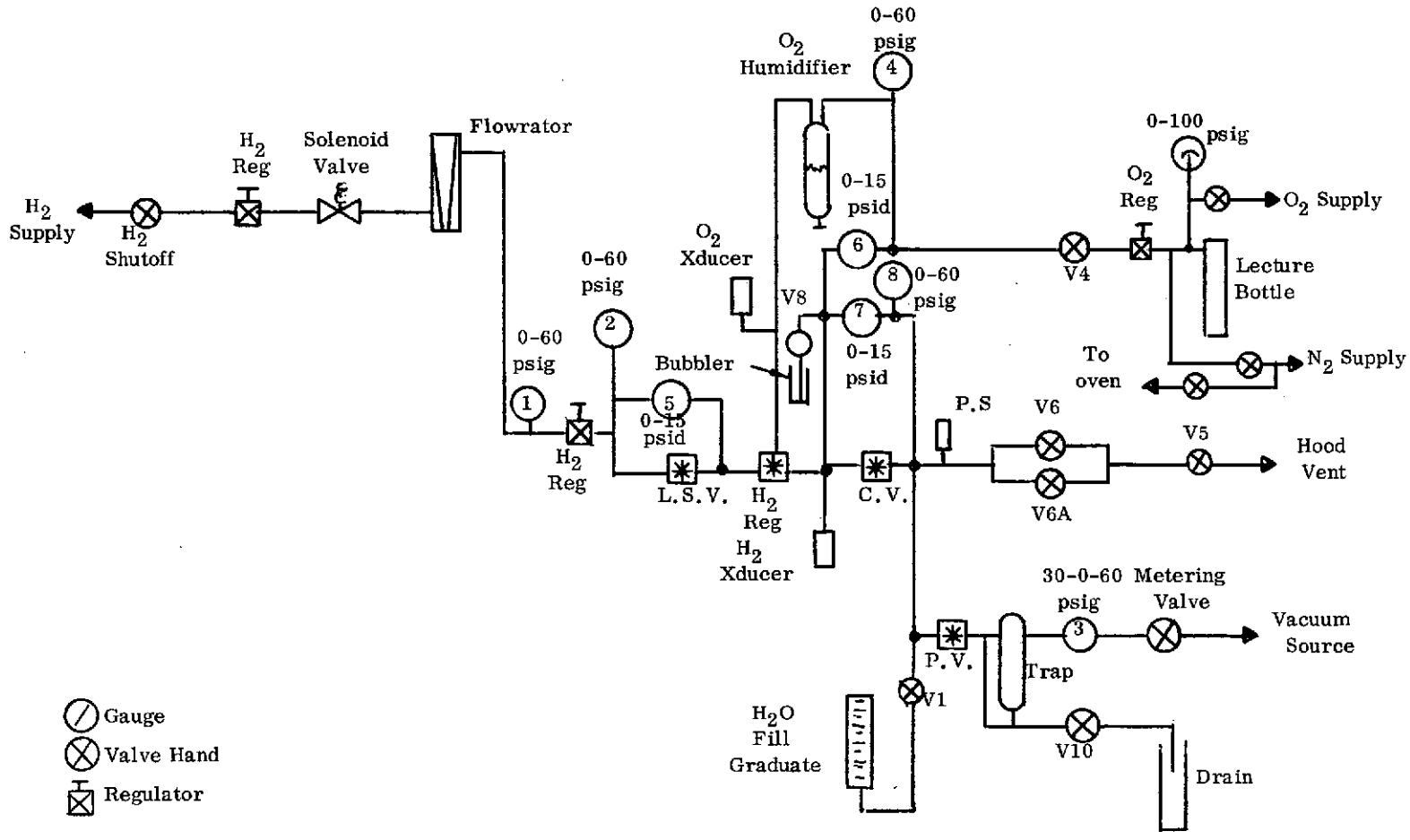
The 180°F and 150°F hydrogen subsystem tests are described in GE/DECP documents SPR-096, dated 11 December 1972, and SPR-048, dated 25 September 1971, respectively.

4.4.1.2 Coolant Subsystem

A fuel cell module coolant subsystem was tested for 2465 hours over 14 simulated missions at 180°F coolant inlet conditions to determine failure rates, materials compatibility and performance characteristics. All of the subsystem components, except the thermal switch and temperature regulating valve, had previously undergone a successful test for 2535 hours at 150°F conditions, for a total of 5000 hours.

The coolant subsystem components tested were:

Coolant pump
Heat exchanger



* All Space Shuttle components mounted in oven - (N_2 ventilated)

Figure 28. Hydrogen Subsystem Life Test (180°F) Schematic

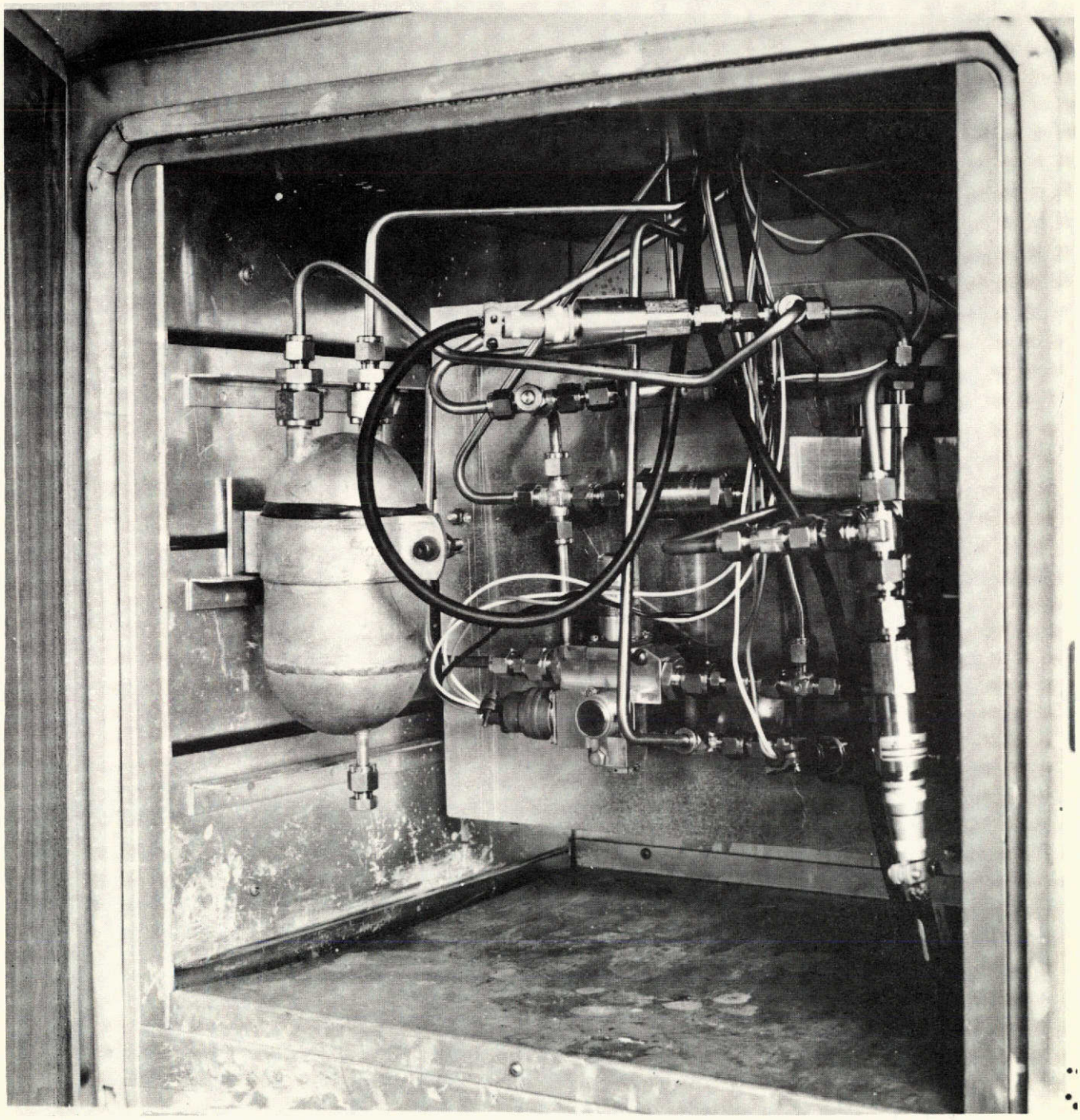


Figure 29. Hydrogen Subsystem Components Mounted in Oven

Coolant accumulator
Transducers (2)
Thermal switch (210°F)
Temperature regulating valve (173 - 183°F)

These components were connected to a fuel cell module simulator containing fuel cell materials with sufficient instrumentation for data acquisition. The components and piping were insulated to maintain 180°F operating temperature. Coolant pump power was provided by a DC to 3-phase AC inverter. Traces of coolant pump differential pressure for Cycles 1 through 14 established the invariant performance of the pump during steady state and transient conditions.

Figures 30 and 31 show the test schematic and the test setup.

Traces of coolant inlet temperature to the simulated fuel cell module for 14 cycles of the load profile demonstrated that the thermal stability of the system was excellent under steady state (2 KW) conditions. During periods of transient testing (1.9, 6.4 and 4.5 KW), the thermal control and recovery were satisfactory at all times. The coolant subsystem test load cycle profile is shown in Table I.

Table I

Coolant Subsystem Load Cycle Profile at 180°F and 35 psig

Time (Approximate)	Load, KW
6 minutes	6.4
15 minutes	2
2-3 hours	Shutdown
30 minutes	2
18 minutes	4.5
165 hours	2

All coolant subsystem components successfully demonstrated suitability for use with the fuel cell module at elevated temperatures (180°F) and exhibited extended life capability (5000 hours) at Space Shuttle mission requirements.

The 180°F and 150°F coolant subsystem tests are described in GE/DECP documents SPR-069, dated 17 March 1973, and SPR-048, dated 25 September 1971, respectively.

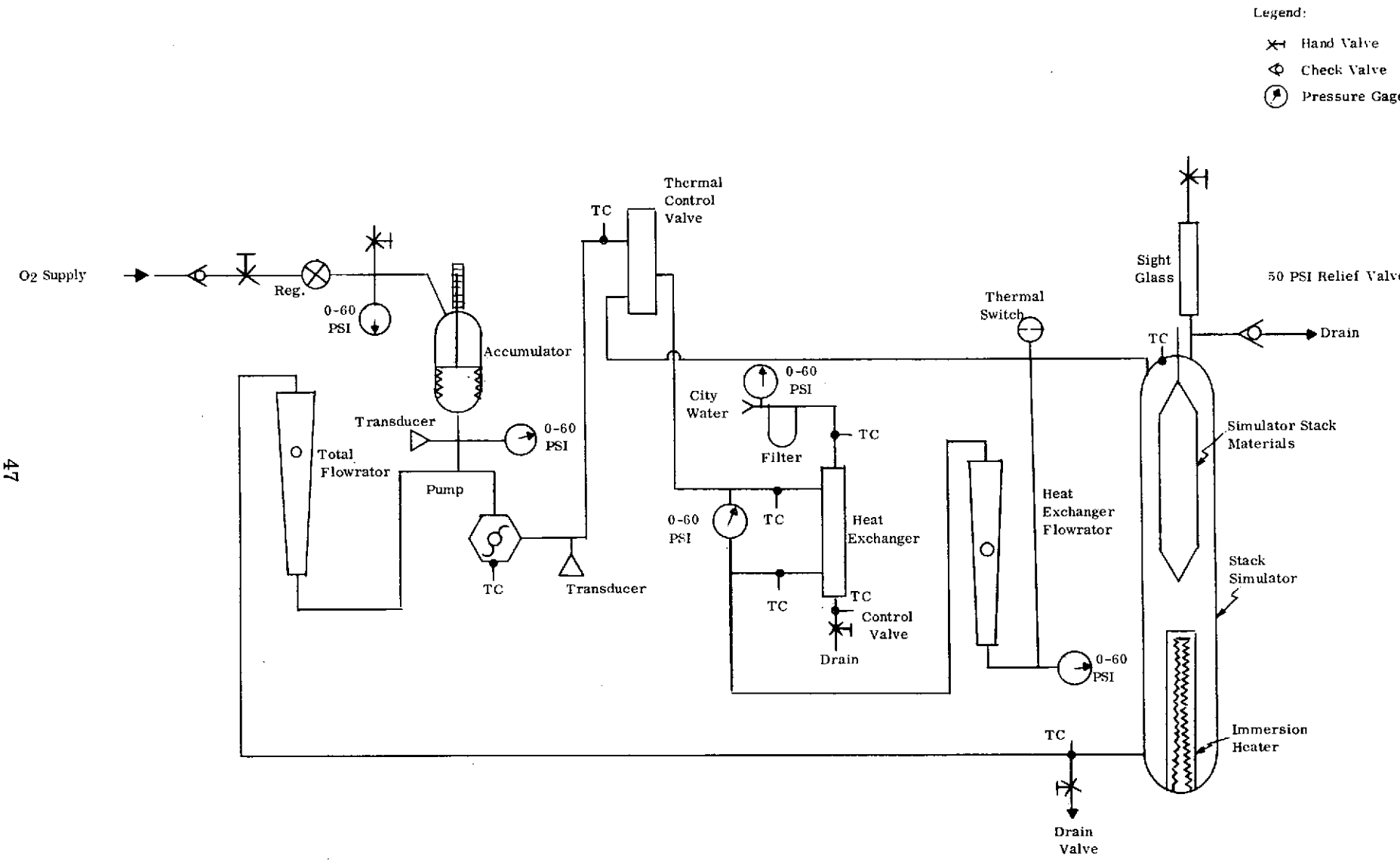


Figure 30. Coolant Subsystem Life Test Schematic

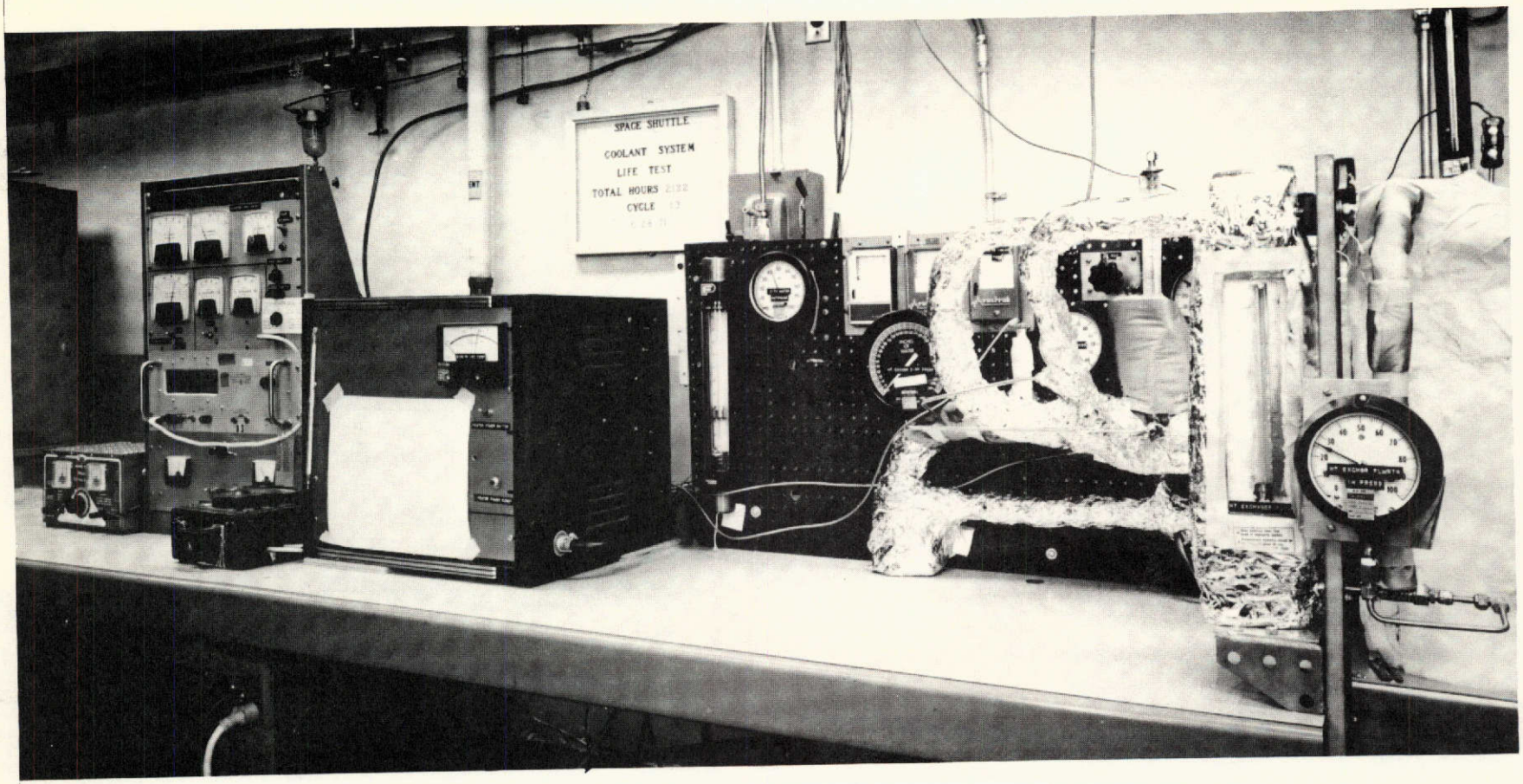


Figure 31. Insulated Coolant Subsystem Components Mounted on Test Bench

4.4.1.3 Prehumidifier, Oxygen and Product Water Subsystem

A fuel cell module reactant prehumidifier, oxygen and product water system was tested for 2488 hours over 12 simulated mission cycles to determine failure rates, materials compatibility, and performance characteristics at 180°F operation conditions. All of the subsystem components, except the reactant prehumidifier and water pressure regulator, had previously undergone a successful test for 2013 hours at 150°F, for a total of 4501 hours.

The subsystem components tested were:

Water pressure regulator
Latching solenoid valve (inlet)
Normally-closed solenoid valve (purge)
Check valve
Transducer
Oxygen and hydrogen reactant prehumidifier

The components were installed in an oven to achieve 180°F operating conditions. Figures 32 and 33 show the test schematic and the components installed in the oven. The reactant prehumidifier was located outside the oven.

Facility and checkout tests were conducted in conjunction with the reactant prehumidifier prototype. Operation of the prehumidifier was generally acceptable, with the exception of a high cooling passage ΔP. Reactant saturation was obtained at less than 2°F temperature drop from the coolant temperature entering the humidifier. Since there is a coolant ΔT of 5°F through the stack at base load operation, the saturated reactants would enter the cell at approximately 3°F above the cell temperature at the inlet area.

The end plate prehumidifier was disassembled for design modifications to reduce coolant ΔP and to install catalyst on the water side of both humidifying membranes to eliminate H_2 in product water.

These changes were incorporated and the unit returned to test. Initial results showed:

a) 50% reduction in coolant ΔP.
b) H_2 humidity dew point 1°F < coolant inlet.
c) O_2 humidity dew point 4°F < coolant inlet.
d) < 1% H_2 in product water evolved gases.

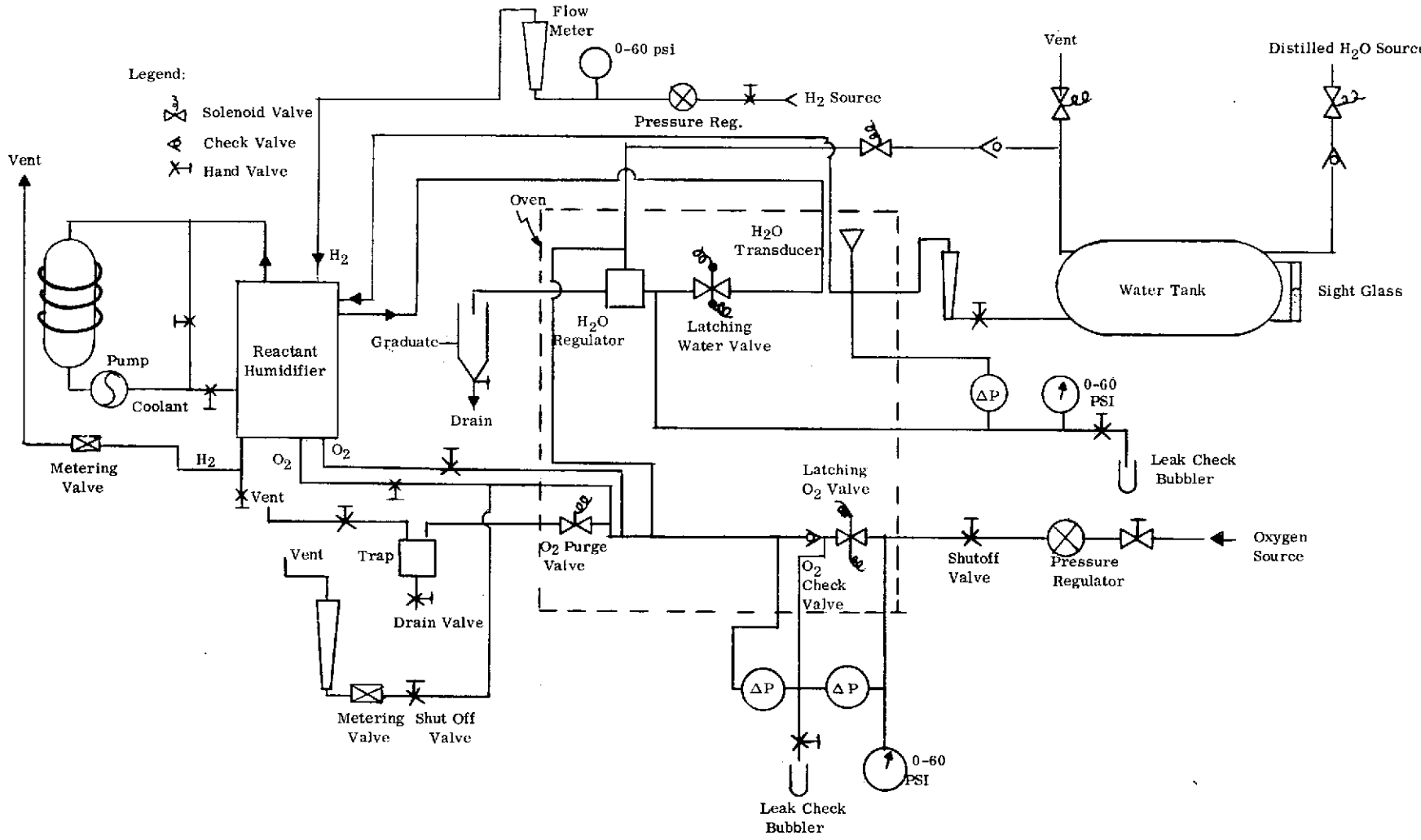


Figure 32. Oxygen and Product Water Subsystem Life Test Schematic

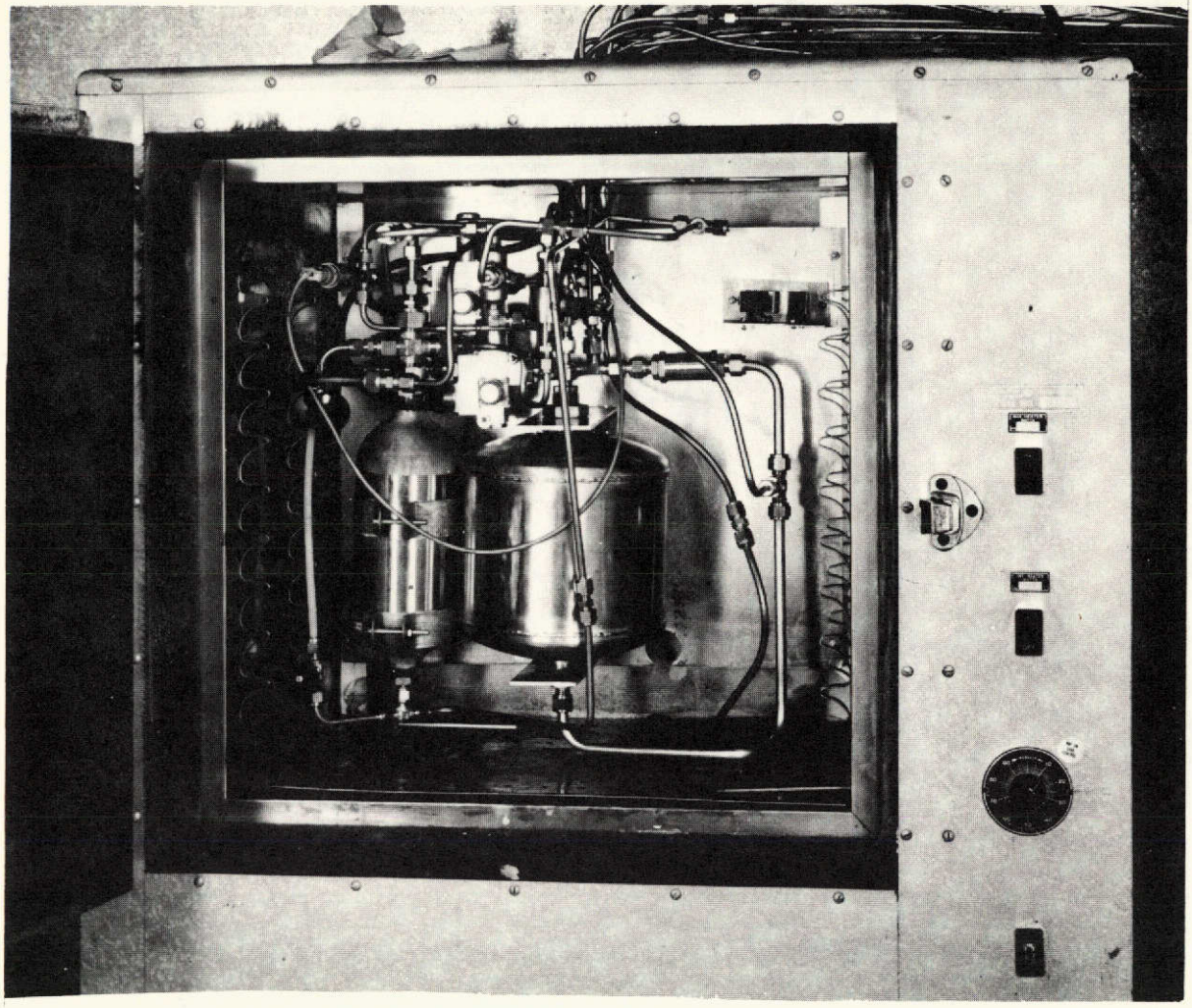


Figure 33. Oxygen and Product Water Subsystem Components Mounted in Oven

The coolant inlet to the prehumidifier is coolant outlet from the cell stack. Since the coolant ΔT across the stack at the base 40 amp load is 5°F, the reactants entering the cell assembly on the cooler side of the cell assembly would be saturated at a temperature slightly higher than inlet area.

The subsystem accumulated a total of 950 hours of operation at 180°F, at which time the product water regulator malfunctioned resulting in a no flow condition. This equipment was replaced with a facility hand valve and returned to the vendor for analysis. Failure was caused by a delamination of the molded silicone rubber valve head.

The testing continued with a facility hand valve simulating the water regulator, and the subsystem accumulated 1550 hours of operation at 180°F.

At the 1627 hour point of 180°F testing, the reworked water regulator was installed into the set-up. Performance was normal. At 1667 hours, the prototype end plate humidifier was removed so as to allow check-out of the EM-1 prehumidifiers. The EM-1 prehumidifier for Stack FS-2 demonstrated all design requirements.

The subsystem accumulated a total of 2488 hours of operation at 180°F. A complete operational checkout of the EM-1 prehumidifier for Stack FS-3 demonstrated all design requirements. Following the FS-3 prehumidifier checkout, this subsystem testing was terminated as having achieved the objective life.

4.4.2 Fuel Cell Container Assembly Tests

4.4.2.1 Container Proof Pressure Test

The titanium fuel cell container, which was originally designed for 22 psia operation in the Gemini and Biosatellite applications, was upgraded for 45 psia operation. See Figure 34. The container assembly was pressure checked to 45 psig (60 psia) and found to be leaktight. Increasing the pressure to 57 psig (72 psia) gave the first indication of leakage at the ring mount seals and the accessory pad seals. Increasing the pressure to 90 psig caused additional gasket seal leakages but no container deformation.

From the standpoint of container stresses during ground testing, the 45 psig point is equivalent to an operating O_2 pressure of 60 psia, and the 90 psig represents at least a 2 times safety factor for burst pressure. The leakage occurring at 57 psig (equivalent to 72 psia of operating O_2 pressure) provided sufficient margin to consider using the remaining containers on hand for the ground testing of EM-1 at 60 psia.

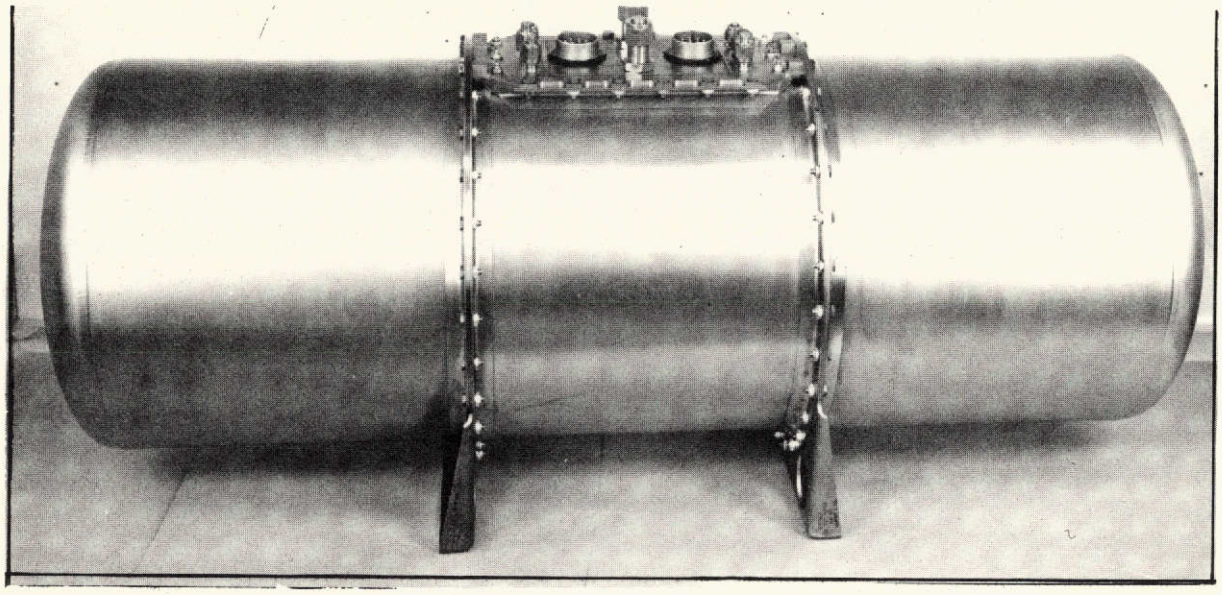


Figure 34. Flight Configuration Container

4.4.2.2 Container Fire Safety Test

The "worst case" condition assumed for the fire safety testing considered three series failures of module components as follows:

1) A leakage of the hydrogen subsystem to oxygen subsystem within the container must develop.

2) A reactant regulator malfunction must occur such that hydrogen pressure is above oxygen, whereas oxygen is normally 4 psi above hydrogen.

3) A failure of the oxygen/hydrogen transducers or electronic readout to sense the hydrogen-over-oxygen condition.

The above series of failures occurring simultaneously has been estimated at 1×10^{-13} occurrences per hour.

Following the proof pressure test, the container was set up for a "worst case" fire safety test. Fuel cell construction materials were installed within the container with a 316 SS tube impinging on a fuel cell electrode catalyst. The container was purged with oxygen gas and set at 45 psig (60 psia). Hydrogen gas, preset at 50 psig, was introduced into the container through the tube impinging on the catalyst. An internal fire was initiated and confirmed by increasing skin temperature as measured by thermocouples and by internal pressure increases. The pressure rose over a 39-second period to about 125 psig, at which time a 3 inch diameter hole burned and/or melted in the titanium container skin, relieving the internal pressure. During the pressure buildup period, leakage of gas was audible. This occurred probably at the gasket sealing areas of the container. However, the leakage rate was apparently not fast enough to completely dump the container pressure as has been the case in previous testing of this container assembly design.

Further analysis of the fire safety test results led to the following conclusions:

a) The titanium material in the container did not contribute to the problem.

b) Available oxygen in the container was much greater than would be the case with installed fuel cells, thus contributing to sustaining the fire.

c) The test may have been unrealistically conservative because of the very small quantity of wet wicking material and lack of a flowing coolant system.

The approach to the "worst case" safety of the container consisted of determining the methods for relieving the generated pressure from the container in the unlikely event that a fire developed outside the fuel cell stacks.

An analysis of the container pressure rise rate was performed, and a pressure relief valve specification was generated from this analysis. Ausco Inc. supplied three valves to this specification. The three container relief valves were acceptance tested for cracking and reseat pressure and for flow rate. All requirements were within established limits.

One relief valve was selected and installed on the Space Shuttle fuel cell module container. A simulated free oxygen volume at 60 psia was established within the container. A fire was initiated by introduction of hydrogen into the pressurized oxygen atmosphere in the presence of a catalyst. The relief valve prevented pressure increases above the container proof pressure level; however, the container relieved to ambient pressure when a hole was melted through the container. Although this test represented a substantial improvement over the previous test, it was not considered adequate for personnel safety under the worst case conditions.

Following additional analysis, three fire safety tests (worst case conditions) were performed in development test cases. These tests differed from earlier tests in that the simulated stack materials were wrapped in two layers of glass cloth and that the container pressure was relieved to ambient with a pressure rise of 10 psi above normal operational pressure. All three tests were successful in that no evidence of fire or burning material penetrated the glass cloth. A piece of titanium container sheet stock laid adjacent to the cloth was untouched by the fire.

The same testing was then performed on the titanium container itself with equally good results. The EM-1A design was then considered as safe for testing in the titanium container with the following features:

a) Stacks wrapped in two layers of glass cloth.

b) Automatic shutdown, including reactant inlets, with an increase in pressure of 5 psi above normal operational pressure via oxygen subsystem transducer and monitoring control unit.

c) Automatic dumping of oxygen and hydrogen subsystems to ambient with an increase in pressure of 10 psi above normal operational pressure via an oxygen subsystem pressure switch and independent valves and circuits (independent of monitoring control unit).

This configuration of a flight-type titanium container was then introduced to endurance test with EM-1A and successfully demonstrated over 2000 hours of operation without mishap, prior to the scheduled test termination.

5.0 MATERIALS TESTING

5.1 Polysulfone Frames

Frames of the configuration shown in Figure 35 were die cut from 0.040 and 0.020 inch polysulfone sheet. The 0.040 inch stock is used as a spacer in the cooling cartridge assembly where the 0.003 inch niobium waffle plates are bonded to each side as shown in Figure 36. The 0.020 inch polysulfone frames are bonded to both sides of the cooling cartridge and subsequently, the membrane and electrode assembly (M and E) is bonded to these frames. All of the bonding is accomplished with an epoxy-nylon adhesive which is cured for 30 minutes at 330°F and 60 psi of clamping pressure. The flow of the bonding material at cure temperature results in a flashing around the inner or outer perimeters of the frame which is normally not removed.

The coolant cartridges fabricated for the first full stack (FS-1) were made with two 0.020 inch frames laminated together, whereas Stacks FS-2, FS-3 and FS-4 were fabricated with single 0.040 inch frames. In all of the investigations into the cause for cracked frames, the center frame was never found to crack. However, cracks in this member would not be visible, and water pressure inside the cartridge is slightly higher than the oxygen side so that cracks of the nature found would likely not be discovered by leak tests of the coolant system.

Full Stack FS-1 has never exhibited any signs of oxygen-to-hydrogen leakage over its 5000 hours of operating life and nearly a year of storage. Therefore, it has never been disassembled for a complete cell assembly examination. When the six cells were removed after 4400 hours, the remainder of the stack was not disturbed. When the six cells were disassembled, they showed no signs of cracks. However, one spare cell assembly made during the same production run did exhibit a crack in the M and E frame.

The cracks occurring in the cells from Stack FS-3 were initially observed along sides A and B as shown in Figure 35. Later, when FS-2 failed and the cell assemblies were examined in detail by stripping the M and E's, cracks were also found along sides C and D. At the time when cell assemblies from FS-4 were examined, cracks were again found in the side C and D areas in the M and E frame.

Tensile and elongation tests were performed on samples of the 0.020 inch thick polysulfone material taken from both directions of the raw material, from samples of cut frames, from old stock, from new stock and from annealed stock. In all cases, the results correlated with materials handbook values of 11,000 to 12,000 psi and 11 to 14% elongation at room temperatures. Also, at 350°F, the tensile was 3,000 to 4,000 psi and the elongation 3 to 6%. The calculation of stresses resulting from differential expansion rates between the polysulfone and the bonding fixtures

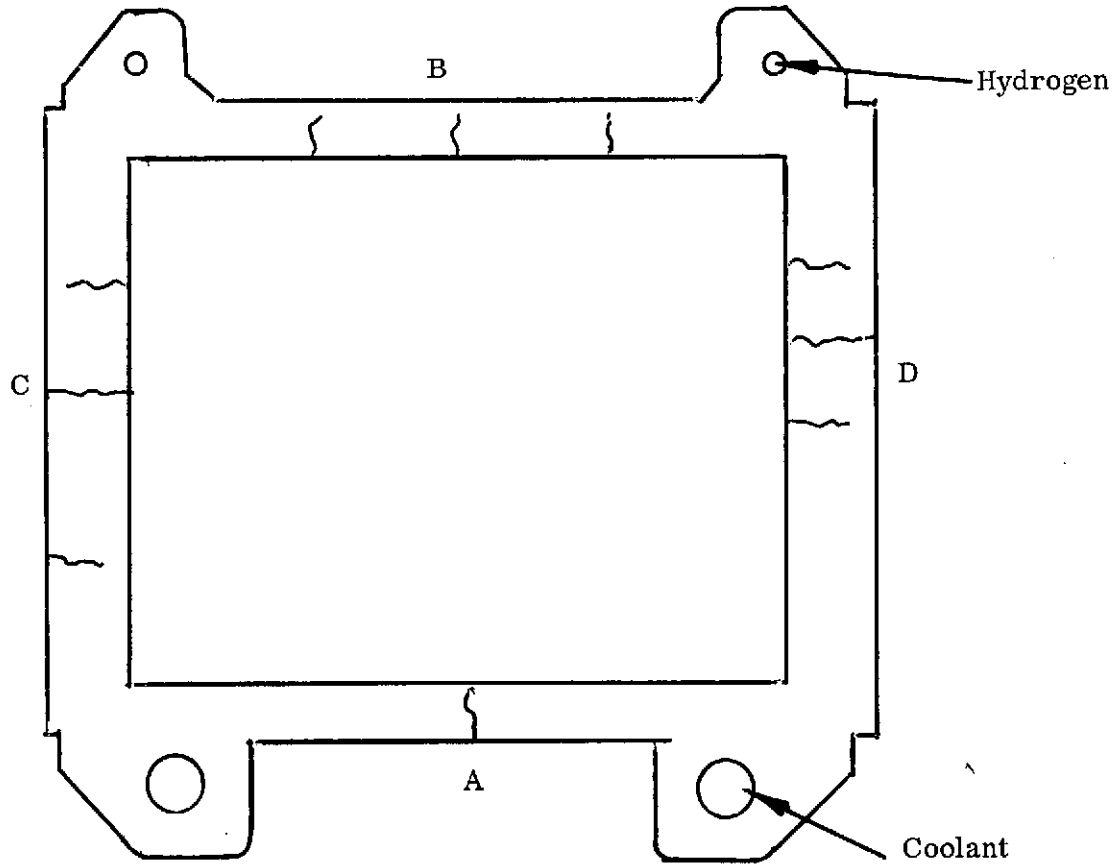


Figure 35. Polysulfone Frame Configuration and Crack Locations

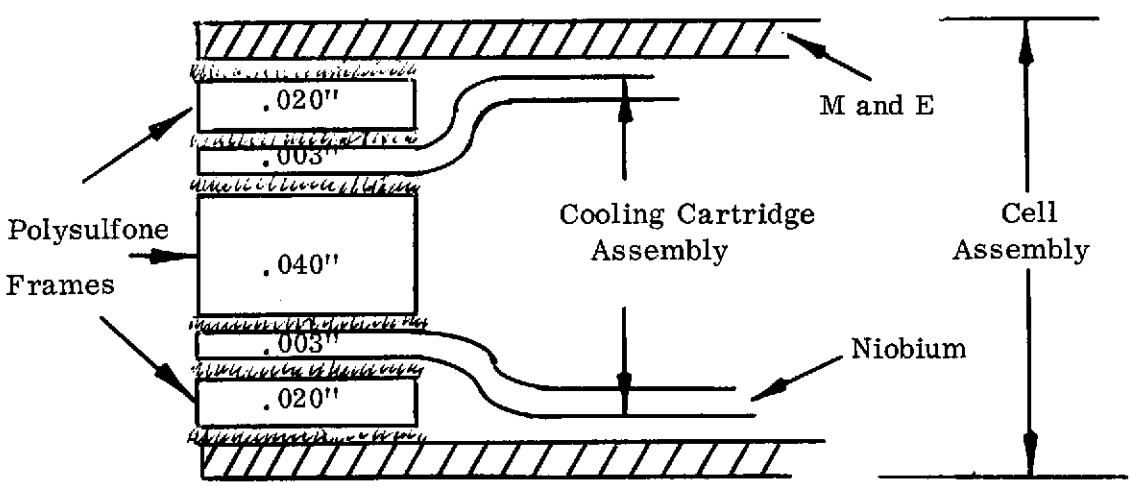


Figure 36. Cell Assembly Cross-section

results in a stress of only 1750 psi if both ends of the part are fixed. However, polysulfone is susceptible to cracking at low strains in the order of 1% at temperatures of 280 to 375°F. Strains in the order of 1.5 to 2% can occur in the bonding operation at 330°F due to the differential expansion rates if the parts are restrained during this process.

Obvious differences in the restraint of the frames during bonding were found between the fixtures used during the fabrication of Stack FS-1 and the subsequent fabrication of Stacks FS-2 and FS-3. The locating pins were closer together in the latter fabrications and the fixtures were not as flat. These conditions were corrected before the fabrication of Stack FS-4. Samples run through the bonding cycle which were restrained at both ends did indeed duplicate the cracking observed in the cell assemblies.

Completed cell assemblies were initially leak tested in air with a vacuum drawn on the hydrogen side (14 psid). Due to the relatively low pressure and because of the diffusion rate of oxygen from the air, it was concluded that this test was not sufficiently sensitive to find the cracked frames. Therefore, the test was changed to 60 psid of nitrogen performed directly after bonding when the cell was the driest. The procedure was applied to all the cell assemblies of FS-4 and to subsequent full stack tests. It was also found that cracks in the M and E frame could be readily seen under a strong slant light. This procedure was also applied to the fabrication of Stack FS-4. After the M and E was bonded, visual examination was not feasible because of the opaque nature of the M and E. Examination by X-ray was not possible because of the low density of the polysulfone. Late in the investigation, a method of neutron radiography was developed by Jet Propulsion Laboratory consultants, but the equipment needed precluded its practical use as a production inspection tool.

It was concluded that the cracks occurred only during the bonding operation and could not result from operational conditions or the effects of stacking. The operational temperature is only 180°F where the strength of the polysulfone is 7000 to 8000 psi, while the stress from stacking is only 600 psi, and the maximum ΔP stressing is only 1250 psi in tensile.

In spite of the fixture changes, the improved visual inspection and the more sensitive leak test method used in the fabrication of FS-4, cracks were observed in these frames during the teardown to repair the plugged manifold in one cell. It was evident that the bonding flash covered the ends of the cracks in the cell assembly so that they could not be seen and they would initially not leak. Unsupported bonding flash will deteriorate with time, causing the ends of the cracks to be exposed and allowing oxygen-to-hydrogen leakage even at the normal operating ΔP of 4 psi. This would account for the random time incidence of cell assembly leaks.

Full stacks FS-2 and FS-4 were patched with RTV 108 silicone adhesive along the edges of the cells after removal of the bond flash. These stacks were returned to test where Stack FS-4 completed 2000 hours of operation and Stack FS-2 successfully operated through a series of special tests as described in the previous section.

The conclusions reached from the analysis of the polysulfone cracking problem are as follows:

1) The susceptibility of polysulfone to cracks caused by very small strain levels at the bonding temperature of 330°F results in a very marginal process to control.

2) An alternate bonding process and a frame material were available which do not require a thermo-setting adhesive. Both the silicone contact adhesive and the Teflon frame material are completely compatible with the solid polymer electrolyte fuel cell.

3) Even though the polysulfone frame in the cooling cartridge has never caused a failure in past testing, it also should be replaced since all of the conditions are present for causing cracks in this part.

5.2 Teflon Frames

The Teflon frame material chosen as a solution to the polysulfone frame cracking problem has been used by GE in solid polymer electrolyte fuel cell assemblies since 1967 with no evidence of material degradation or failure. The material as used in the B/U 106 cells consists of a 0.006 inch thick sheet of Teflon with 0.004 inch of a silicone rubber contact adhesive applied to each side of the Teflon. The material is fabricated by Technical Fluorocarbon Engineering, Inc., of Warwick, Rhode Island. This material was not initially considered for the Space Shuttle Fuel Cell configuration since it results in a 5 lb weight increase for a 10 KW unit. Further, there was a reluctance to change from a bonding system developed prior to this Technology Program that appeared to be satisfactory.

The life testing of B/U 101 indicated that the AF-42 bonding material had lost most of its adhesive strength after 2000 hours of operation. The continued cell integrity for the next 4500 hours of life was dependent on uniform compression pressure from the oxygen side gasket assembly. Performance evaluations had also identified a contaminating constituent in the AF-42 adhesive that was causing a loss of 30 to 40 millivolts at the cathode electrode. Evaluation of alternate adhesive systems revealed that the silicone rubber contact adhesive provided an adequate bond for the cell assemblies as well as recovering 25 to 30 millivolts of the anticipated performance.

A notable example of the successful application of the silicone adhesive/Teflon frame material is a 4-cell development test unit (AFC-6), which has operated for more than 22,000 hours at 180°F. AFC-6 has operated without maintenance and with exactly the volt/amp performance at 22,000 hours as at the start of test. In addition, small stack buildups B/U 105 and 106 of Space Shuttle prototype cell assemblies, as described in para. 4.1.5 and 4.1.6, have also been life tested using the silicone adhesive/Teflon frame material. B/U 106 has successfully operated for over 2000 hours at this time.

An extensive series of laboratory evaluation tests has been completed on the silicone adhesive/Teflon frame material including creep tests, water aging tests, auto-ignitions, leaching tests and hydrogen peroxide degradation tests.

A creep of 8% was observed after 2000 hours at a constant loading of 400 psi at 220°F. This is well within the capability of the RTV gaskets to compensate for this creep and still provide sufficient sealing pressure. The cell assembly contains two RTV gaskets having a total thickness of 0.170 inch, and three Teflon frames having a total thickness of 0.034 inch. The gaskets are assembled with a 10% compression, while a 5% compression is adequate for sealing. Thus, 5% of 0.170 inch or 0.0085 inch of creep can be accommodated in the cell assembly design. The observed 8% creep of the Teflon results in a dimensional change of 0.0027 inch. Furthermore, an extrapolation of the Teflon creep data to 10,000 hours would indicate a creep of 16% or a 0.0054 inch dimensional change, which is still well within the gasket capability.

The water aging tests at 200°F showed no loss of bond adhesion over 1000 and 2000 hours for the membrane-to-frame and niobium-to-frame samples, respectively. The observations during the teardown of B/U 105 and B/U 106 confirmed the creep and bond integrity data.

Auto-ignition tests in 100 psig oxygen showed ignition at 485°F, which is consistent with published literature for both silicone rubber and Teflon. Both materials are already used in other parts of the cell assembly.

Samples of the silicone rubber adhesive/Teflon frame were leached in .001 $\underline{N}$ H_2 SO_4 at 70°C for 500 hours. No detectable inorganic material was leached from the samples, and a just barely detectable amount of organic material was extracted.

In the hydrogen peroxide degradation test, the samples were treated in 30% H_2 O_2 and 1 mg of Fe at 80°C. No release of fluoride was ever detected under these conditions.

The conclusions drawn from this material evaluation are that the silicone rubber adhesive/Teflon frame is completely compatible with the fuel cell environment for more than 2000 hours. The life demonstrations in fuel cell hardware confirm the acceptability of this material as an alternate to the polysulfone frame with the epoxy-nylon adhesive system.

5.3 Materials Flammability

A Materials Flammability Report (SPR-071, dated 15 March 1972) was submitted to NASA/JSC for review and approval. This report included the complete materials list for the baseline Fuel Cell Module designed for a 5 KW output, and which consisted of two stacks of 40 cell assemblies each at that time.

A memorandum from J. H. Kimzey (ES-5) to G. D. Hydrick, Jr. (EP-5) at NASA/JSC, documents the review of this report by the Structures and Materials Division at NASA (see attached copy). Since this report was written, the wicking material has been changed from Dacron to Refrasil glass cloth, and the ethylene propylene rubber, nylon and the AF-42 adhesive have been replaced. Thus, all of the materials now used in the Solid Polymer Electrolyte Fuel Cell are considered as acceptable for use in this application.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS 77058

REPLY TO ATTN OF: ES5

May 26, 1972

MEMORANDUM

TO: EP5/George D. Hydrick, Jr.

FROM: ES5/J. H. Kimzey

SUBJECT: GE Shuttle Fuel Cell

A review of GE Report SPR-071 dated 15 March 1972 was made this date. The following is noted:

1. From the standpoint of environmental effects on materials, the use of "psig" is meaningless. We need absolute pressure, psia. If the value is 50 psig for oxygen, the reference may be "atmosphere," "vacuum," or the gaseous hydrogen, giving values of 65, 50, or 96 psia according to the interpretation.

2. For a temperature of 150°F in oxygen at 65 psia and dry, except for 100 percent relative humidity, there are many nearly noncompatible polymeric materials. The use of dacron as a wick assembly etc., is probably a worst case since over 170 square feet are listed. The use as a wick, however, implies liquid water coating most of this material. Other particularly vulnerable material includes ethylene propylene rubber (Table II), nylon, and various adhesives.

3. As temperatures and pressures increase such as to 180°F and 75 psia the condition is more marginal. The actual ignition of such materials at these pressures has never been studied particularly with various aging aspects also considered.

4. Without a further temperature increase, electric arcing, or mechanical shock, we can say that these materials will not ignite, but the potential energy is obviously very high. For worst case conditions such a fire would be oxygen limited. There is a significant excess of fuel.

J. H. Kimzey

ES5/JHKimzey:tsh:5/26/72:3561

6.0 SIGNIFICANT ACHIEVEMENTS

The accomplishments achieved during the course of this contract are summarized as follows.

(1) Demonstrated that the operating life capabilities of the Solid Polymer Electrolyte Fuel Cell on prehumidified reactants exceeds 5000 hours. The fuel cell performance is invariant, with life with the efficiency and specific reactant consumption unchanged from start to end of life. Also, the extremely simple stop and start procedures with simulated Space Shuttle mission profiles have been satisfactorily demonstrated. A summary of the significant tests performed is as follows:

o	>20,000 Hours,	4-cell stack,	Boilerplate hardware, continuous operation.
o	6,500 Hours,	3-cell stack,	Space Shuttle hardware, 50 stop/start cycles.
o	5,000 Hours,	38-cell stack,	Space Shuttle hardware, 32 stop/start cycles.
o	4,300 Hours,	4-cell stack,	Space Shuttle hardware, 28 stop/start cycles.
o	2,000 Hours,	34-cell stack,	Engineering Model (EM-1), 8 stop/start cycles.
o	>2,200 Hours,	2-cell stack,	B/U 106 Improved Baseline, 19 stop/start cycles.

(2) Demonstrated the operating life capabilities of the Fuel Cell Module Ancillary Components. The significant tests performed were as follows:

- o 5000 hours on the coolant loop components.
- o 4000 hours on the reactant and product water loop components.
- o 5000 hours on all components as part of test facilities supporting the full stack testing.
- o 2000 hours on all components as part of the integrated EM-1 Module.
- o 2000 hours on the end plate integrated prehumidifier as part of the EM-1 Module.

(3) Demonstrated scale-up capability both for numbers of cells in a stack and for cell active area. The full stack of 34 to 38 cell assemblies produced the same performance and life capabilities as the small stack of 3 to 4 cell assemblies of the same configuration and operating conditions. Also, the scale-up in active cell assembly area from 0.70 ft^2 to 0.82 ft^2 produced the predicted performance and has operated with stable performance for over 2200 hours to date.

(4) Demonstrated the solution to the problem of the polysulfone frame cracking problem by substitution of a Teflon frame material with a double-sided silicone rubber contact adhesive. The frame cracking with the polysulfone material and the high-temperature adhesive system was a manufacturing process control problem which affected the life testing of the Engineering Model (EM-1). In spite of this inherent problem, the EM-1 module operated successfully for 2000 hours, demonstrating the ease of maintainability inherent in the solid polymer electrolyte fuel cell and the capability of all the other module hardware to successfully operate for at least 2000 hours as an integrated system.

(5) Demonstrated the safety of operation under the worst case conditions of a triple failure in the fuel cell module container. All of the safety design features operate to isolate the affected module from the remainder of the system to eliminate single point failure modes from affecting the electrical power system.

(6) Demonstrated the most practical method for prehumidification of the reactant gases with a device built into the end plates that did not require any external interfaces and did not require any parasitic power.

(7) Demonstrated quality of product water by reducing presence of fluoride ions to less than 1 part per million. Water, as delivered from the fuel cell, meets all of the potability requirements of NASA Specification SD-W-0020, except for pH. The pH of the water was 4.5 to 5 as delivered and demonstrated to be buffered to 6 or 7 when passed through a small deionizer column.

(8) Demonstrated a new baseline coolant cartridge configuration (B/U 106) incorporating a weight reduction in the hardware of approximately 30% and a reduction in the cooling fluid in the system of approximately 70%.

(9) Demonstrated the capability of the solid polymer electrolyte fuel cell to operate on propulsion grade reactants without the need for any scrubber. Also demonstrated the elimination of the H_2 purge during operation. The SPE is not affected by the normal contaminants in the propulsion grade reactants.

(10) Demonstrated the volt/amp performance levels used as the baseline design for sizing the Fuel Cell Powerplant for the Space Shuttle proposal.

(11) Demonstrated low-cost, completely automated life test facilities, using the electronic breadboard circuitry also adaptable to Automatic Monitoring and Control in the spacecraft operation.

(12) Demonstrated extremely simple start/stop and storage procedures that do not require any inert gases.

- o To Stop - Remove load, shut off hydrogen supply valve and coolant pump. Time - instantaneous.
- o To Start - Turn on coolant pump, open hydrogen vent valves to vacuum for 10 seconds, open hydrogen supply valve and switch on load.
- o Alternate Start - Turn on coolant pump, apply power to unit to electrolysis operation for 10 minutes, open hydrogen supply valve and switch on load.
- o To Store - Same as to Stop.
- o Open circuit - Idle with only coolant pump load indefinitely.

7.0 CONCLUSIONS

The objectives of the Hydrogen-Oxygen Fuel Cell Technology Development Program have been successfully accomplished.

A proven baseline Fuel Cell Powerplant configuration for the Space Shuttle Orbiter is available in the General Electric Solid Polymer Electrolyte Technology. Maintenance-free life of more than 5000 hours with no degradation in performance is well within the capability of this technology.

The General Electric Company, Direct Energy Conversion Programs, has demonstrated the technical and management capability to cope with and solve development problems and to continue improvements in the state of the art.