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INTERIM REPORT

LONG-LIFE, HIGH-PERFORMANCE FUEL CELL PROGRAM

BY

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NASA LEWIS RESEARCH CENTER
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FOREWORD

This Interim Report describes the work completed under the Long-Life, High-Performance Fuel Cell Program, NASA Contract No. NAS3-22234 from 28 May 1981 through 31 October 1984 by the Power Systems Division of United Technologies Corporation.

The NASA Project Manager for this contract was Mr. Dean W. Sheibley. The contributions of Mr. Sheibley and other members of the Electrochemistry Branch Staff at NASA - Lewis Research Center are gratefully acknowledged.

The Project Manager for Power Systems Division was Mr. Ronald E. Martin. Power Systems Division personnel who directed the tasks performed in this program included Mr. W. F. Bell, Dr. J. Powers, and Dr. D. A. Landsman.

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ABSTRACT

United Technologies Corporation has been conducting a multi-task experimental program sponsored by Lewis Research Center of NASA directed toward advancing the state-of-the-art of alkaline electrolyte fuel cells. The goal of the program was the development of an extended endurance, high performance, high efficiency fuel cell for use in a multi-hundred kilowatt Regenerative Fuel Cell for use in a future orbiting Space Station.

A design definition study of a Regenerative Fuel Cell energy storage system was conducted. The study identified system weight, volume, efficiency and an approach to system reliability. Pictorial representations of the system were prepared. The results of study are reported separately under NASA CR-174802.

This technology advancement program has identified a low-weight cell design and cell components with increased performance and endurance. Three 0.508 ft^2 (471.9cm^2) active area multi-cell stacks were assembled and endurance tested at cell temperatures between 140°F (60°C) and 180°F (82.2°C), current densities to 400 amps/ft^2 (430.6mA/cm^2) and 60 psia (41.4N/cm^2) reactant pressure. These full-size endurance tests have demonstrated (1) the long-term performance stability of the platinum-on-carbon catalyst configuration, (2) the suitability of the lightweight carbon electrolyte reservoir plate, (3) the stability of the free standing butyl bonded potassium titanate matrix structure, and (4) the long-life potential of a hybrid polysulfone cell edge frame construction.

A 18,000-hour demonstration test of multi-cell stack to a continuous cyclical load profile was conducted. A total of 12,000 cycles was completed, confirming the ability of the alkaline fuel cell to operate to a load profile simulating Regenerative Fuel Cell operation. An Orbiter production hydrogen recirculation pump employed in support of the cyclical load profile test completed 13,000-hours of maintenance free operation.

Laboratory research endurance tests at 200°F (93.3°C) and 200 amps/ft^2 (215.3 mA/cm^2) has demonstrated the suitability of the butyl bonded potassium titanate matrix, perforated nickel foil electrode substrates, and carbon-ribbed substrate anode for use in the alkaline fuel cell.

Corrosion testing of materials at 250°F (121.1°C) in 42% wgt. potassium hydroxide has identified Ceria, Zirconia, Strontium Titanate, Strontium Zirconate and Lithium Cobaltate as candidate matrix materials.

I. SUMMARY

This interim report documents the activity and results of a multi-year analytical and experimental program to improve endurance capability and performance and reduce weight of hydrogen-oxygen alkaline electrolyte fuel cells for a low earth orbit Space Station application. Work was carried out at the laboratory level on cell components and in fuel cell evaluation tests. Long-term endurance tests of full-size advanced technology multi-cell stacks at both steady and cyclical load profiles were conducted. A Regenerative Fuel Cell system design study was completed and is reported in the separate cover (NASA-CR-174802).

Objectives

The primary development objectives of the program were to:

- Develop a cell separator (matrix) structure which is capable of operating 20,000-hours in an alkaline fuel cell operating on hydrogen and oxygen at 60 psia (41.4 N/cm^2) reactant pressure and 250°F (121.1°C).

- Develop lower cost electrodes which have the potential to operate for 20,000-hours in a hydrogen-oxygen alkaline fuel cell operating at 60 psia (41.4 N/cm^2) reactant pressure and a cell temperature of up to 250°F (121.1°C).

- Endurance test advanced technology six-cell stacks to a cyclical load profile consisting of 60-minutes at open circuit followed by 30-minutes at 200ASF (215.3 mA/cm^2), with a program goal of 20,000-hours of testing.

- Provide program support for the integration, checkout and initial performance demonstration of the Orbiter X-708 fuel cell powerplant as part of the alkaline breadboard energy storage system.

Results

A design definition study was conducted to identify design issues related to the development of a multi-kilowatt Regenerative Fuel Cell System (RFCS) for use in a future space station operating in low earth orbit.

RFCS characteristics were determined for module power ratings of 10kW to 50kW as a function of efficiency. For a 100kW space station, with 33kW modules the RFCS specific weight and volume are 19.9-23.2 watt-hours/pound (43.8-51.1 watt-hours/kg) and 278-283 watt-hours/cubic foot (9817-9994 watt-hours/cubic meter), respectively, at an overall charge-discharge efficiency of 50 percent. The corresponding values for a 55 percent efficiency are 15.9 - 19.3 watt-hours/pound (35.1-42.5 watt-hours/kg) and 258 - 270 watt-hours/cubic foot (9111-9535 watt-hours/cubic meter).

A six-cell stack of 0.508 ft^2 (471.9 cm^2) active area cells was endurance tested to a continuous cyclical load profile consisting of 60-minutes at open circuit followed by 30-minutes on load in a simulation of a Regenerative Fuel Cell load profile. A total of 18,054 hours of operation was completed at a stack coolant inlet temperature of 140°F (60°C). Test results showed improved performance stability with a performance fall-off with time of less than 4 microvolts per hour throughout the test.

Two, 0.508 ft^2 (471.9 cm^2) active area advanced technology four-cell stacks completed 3,500 and 3,000-hours of operation respectively. The performance of cells featuring platinum-on-carbon catalyst anodes, lightweight carbon electrolytes reservoir plates, bonded potassium titanate matrices and hybrid polysulfone edge frames were stable, exhibiting no significant change in performance during endurance testing.

Fine particulates and organic membranes were evaluated as candidate matrix materials with respect to corrosion resistance, manufacturability, and cell operation.

Corrosion testing of materials at 250°F (121.1°C) in hot aqueous potassium hydroxide has identified Ceria, Zirconia, Strontium Titanate, Strontium Zirconate and Lithium Cobaltate as candidate matrix materials. The successful fabrication of bonded particulate matrices from these materials was limited because of cracking and low bubble pressure. However, a laboratory research cell incorporating a butyl bonded strontium titanate matrix successfully completed a planned 5000-hour test.

Porous Nafion®, polyantimonic acid and commercial-grade polybenzimidazole were evaluated as candidate matrix materials. Laboratory research cells incorporating these materials as matrices exhibited rapid voltage fall-off with time.

In previous work, potassium titanate (PKT) was shown to be very stable in hot aqueous potassium hydroxide, PKT samples exhibited less than 7% of the weight loss of asbestos in 250°F (121.1°C), 42% wgt KOH. In this program, high strength, high bubble pressure, "mat-type" matrix was developed by bonding PKT with butyl rubber. Full-size potassium titanate matrix with bubble pressures in excess of 30 psi (20.7 N/cm²) have been successfully endurance tested. Full-size cells incorporating butyl bonded PKT matrices have shown a 60 percent reduction in electrolyte carbonation compared to asbestos and a 70 percent reduction compared to advanced asbestos.

Porous carbon electrolyte reservoir plates have been shown to be stable in the anode environment. This stability was demonstrated in laboratory research cells and multi-cell stack endurance tests. Incorporation of the carbon electrolyte reservoir plate will reduce the weight of a standard production cell by nearly fifty percent.

The hybrid, polysulfone cell edge frame has demonstrated reduced corrosion rates such that electrolyte carbonation with time was reduced by 40 percent compared to standard fiberglass/epoxy frames. The difficulty of achieving uniform edge frame structures has limited the use of the structure in multi-cell stacks. Further development of this concept is required.

Polyphenylene Sulfide (PPS) has been identified as a candidate cell edge frame material. Samples of PPS after 1000 hours of corrosion testing in 250°F (121.1°C) have shown a negligible weight loss compared to fiberglass/epoxy samples with a 20 percent weight loss.

Laboratory research cell endurance testing of cells incorporating gold-plated nickel photo-fabricated foil substrates has shown that the foils are a viable replacement for the expensive gold-plated fine-wire nickel screen currently employed. The cells completed over 4300 hours of endurance testing.

Laboratory research cells incorporating supported platinum catalyst anodes have demonstrated the potential for long-life during endurance testing. Laboratory test results provided the confidence to incorporate the anode catalyst into the long-term six-cell stack which completed 18,054-hours of testing.

Twenty-four laboratory research cells incorporating carbon ribbed substrate anodes, with the potential to reduce cell weight and cost were endurance tested. Test results indicate the carbon ribbed substrate anode concept is feasible.

Long-term endurance testing of five research cells incorporating bonded potassium titanate matrices has demonstrated the stability of matrix configuration at temperatures up to 250°F (121.1°C).

The ribbed nickel substrate cathode which has the potential to improve operational reliability was endurance tested in laboratory research cells and the substrate was shown to be stable at cathode potentials.

Research cell endurance testing has indicated that a selectively wet-proofed anode configuration improves cell tolerance to electrolyte volume variations resulting from changes in operating conditions.

Conclusions

- The ability of the alkaline fuel cell to operate in a cyclical mode, representative of a Regenerative Fuel Cell Energy Storage System (RFCS) was demonstrated. Cyclical testing has shown the potential of the platinum-on-carbon catalyst anode for extended endurance.

- The lightweight carbon electrolyte reservoir plate (ERP) was shown to be suitable for use at the anode of the alkaline fuel cell.

- A high bubble pressure corrosion-resistance, bonded potassium titanate matrix was developed. The matrix configuration has the potential to extend cell endurance by reducing the quantity of corrosion products that accumulate in the cell.

- A new cell edge frame which has reduced contribution to electrolyte carbonation is required. Corrosion-resistant frame materials for reducing carbonate formation and extended cell endurance capability have been identified.

- Improved alkaline fuel cell components were identified from laboratory research cell endurance tests. These components include the carbon ribbed substrate anode and the perforated nickel foil substrate which offer benefits of low-weight, low-cost, and improved reliability.

II. INTRODUCTION

Background

United Technologies Corporation has maintained an active program of fuel cell research, development, production and delivery since 1959. The activity includes fuel cells which use phosphoric acid or molten carbonate electrolyte and fuel cells which employ alkaline electrolyte. The alkaline electrolyte fuel cells operate on hydrogen and oxygen and have been developed for aerospace and undersea applications. The current status of the twenty-five years of research and development is represented by the 12kW fuel cell powerplant shown in Figure 1 for the Space Shuttle Orbiter and a 30kW unit for the U.S. Navy.

The development history of alkaline electrolyte fuel cells and the technology advances from continuing technology programs sponsored by NASA at United Technologies Corporation is presented in Appendix B.

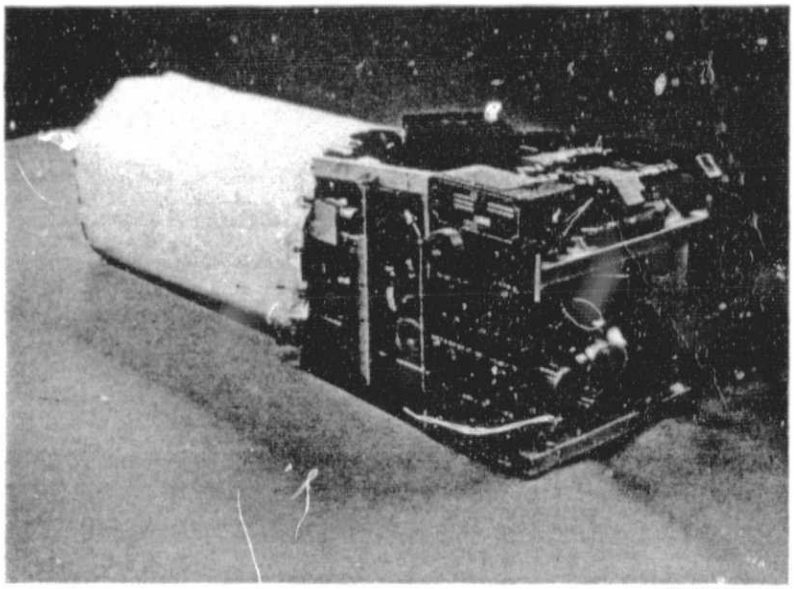

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Figure 1. Orbiter 12-kW Fuel Cell Powerplant

Program Scope

The work being done under this contract continues the National Aeronautics and Space Administration - Lewis Research Center sponsored program to advance the state-of-the-art of space power alkaline electrolyte fuel cell technology. The continuing program establishes a broad-based technology from which complete fuel cell systems could be developed for application to future space missions. The emphasis of the present program is to develop a high performance, high efficiency fuel cell for application in an orbiting space station in which very long-life is the primary design goal.

Facilities and Test Conditions

During the past twenty years, United Technologies has established facilities and assembled equipment to conduct research, development and manufacture of fuel cells. These facilities are presently being used to manufacture 12kW Space Shuttle Orbiter units and other units up to 4.8 mW for Government and Commercial customers. Complete fuel cell powerplants up to 1 mW class can be tested with existing equipment. These facilities are housed in a 200,000 square foot building located in South Windsor, Connecticut.

Under the program, improved cell components were identified and incorporated into the alkaline fuel cell and evaluated. The evaluation tests are initially conducted in subscale laboratory research cells. Based upon laboratory test results, the most promising components are incorporated into 0.508-ft^2 (471.9cm^2) active area multi-cell stacks for long-term endurance evaluation.

Multi-cell stack endurance tests were conducted at temperatures up to 200°F (93.3°C) at reactant pressure of 60 psia (41.4 N/cm^2) and at current densities up to 400 ASF (430.6 mA/cm^2). A six-cell stack was endurance tested to a continuous cyclical load profile consisting of 30-minutes at 200 ASF (215.3 mA/cm^2) followed by 60-minutes at open circuit.

The multi-cell stack endurance tests were conducted in test facilities originally built for the Apollo fuel cell program and continuously modified to meet the requirements of the present cells. During the program, two test stands were completely refurbished to improve operating reliability, modernize facilities and upgrade the test facility to handle long-term endurance testing of multi-cell stacks to a cyclical load profile.

Exploratory evaluation of candidate cell components was conducted in subscale 2 x 2 inch (5.08 x 5.08 cm) active area laboratory research cells at cell temperatures up to 250°F (121.1°C) and atmospheric pressure.

Cell endurance tests were conducted employing dynamic water removal, that is cell product water was removed by recirculating hydrogen.

III. STACK EVALUATION TESTING

The multi-cell stacks endurance tested during the program incorporated 0.508 ft^2 (471.9 cm^2) active area cells with a planar dimension of 8.55 x 8.55 inches (21.7 x 21.7 cm). The stacks are designed to operate with dynamic water removal, that is, product water is removed from the cells within the stack by recirculating hydrogen.

The multi-cell stacks consisted of four or six unitized electrode assemblies connected electrically in series through three different types of separator plates.

Each unitized electrode assembly consists of an anode and cathode separated by a electrolyte containing matrix with a porous electrolyte reservoir plate adjacent to the anode. All these elements are unitized into a structural frame which is fabricated of a plastic reinforced fiberglass structure. The unitized electrode assembly is shown in Figure 2.

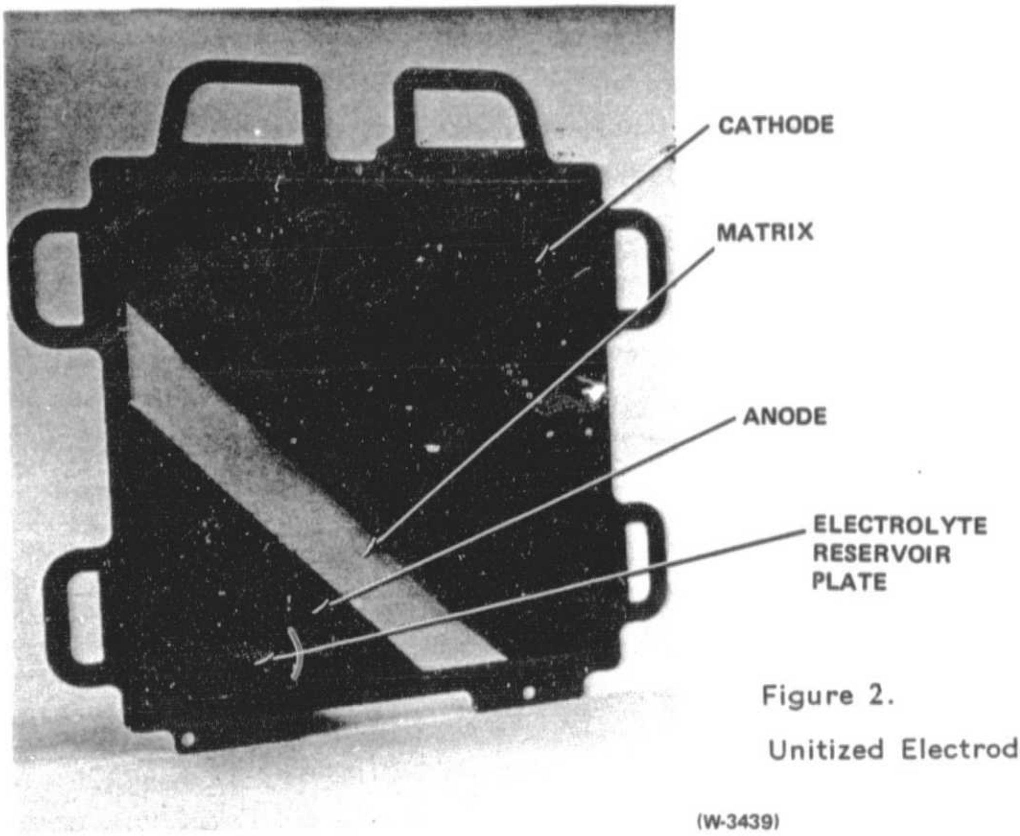

Figure 2.
Unitized Electrode Assembly

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In the multi-cell stack, there are three types of separator plates, a combination plate, a hydrogen plate, and an oxygen plate. These plates are machined out of magnesium sheets for light weight and then gold-plated to insure good contact resistance and good corrosion characteristics. These plates contain flow fields, which provide for the circulation of hydrogen, oxygen and coolant. The unitized electrode assemblies and separator plates are constructed with internal manifolds. Metering ports are machined in the plates so that there is a uniform distribution of hydrogen and oxygen and coolant in the respective flow fields of each cell. The components of the multi-cell stack are presented in Figure 3.

A combination separator plate is installed between every other anode and cathode. The metering ports on one side of the separator plate admits hydrogen to the anode and on the other side of this plate, metering ports admit oxygen to the cathode. Between the alternate cells there are two separator plates, an oxygen plate and a hydrogen plate. The oxygen plate has metering ports which admits oxygen to the adjacent cathode. The hydrogen plate has metering ports in one side which admits hydrogen to the anode and metering ports on the other side to admit coolant between the oxygen plate and hydrogen plate. The separator plates contain molded seals which seal the spaces between separator plates and unitized electrode assemblies so that there is no mixing of hydrogen and oxygen or coolant and no leakage of these fluids from the stack.

The unitized electrode assemblies and separator plates are assembled and compressed between 3/4 inch (1.9 cm) thick stainless steel end plates by titanium tie rods. The end plates are electrically isolated from the cells and separator plates by a non-conducting plastic insulator plate. The separator plates at each end of the stack contain special extensions for attachment of positive and negative load cables. A multi-cell stack setup for performance testing is shown on Figure 4.

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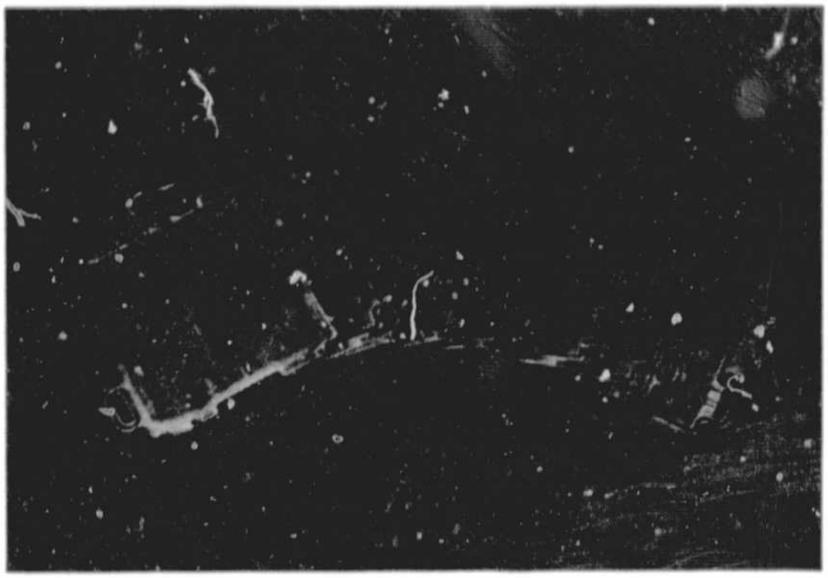

Figure 3. Multi-Cell Stack Components (WCN-9739)

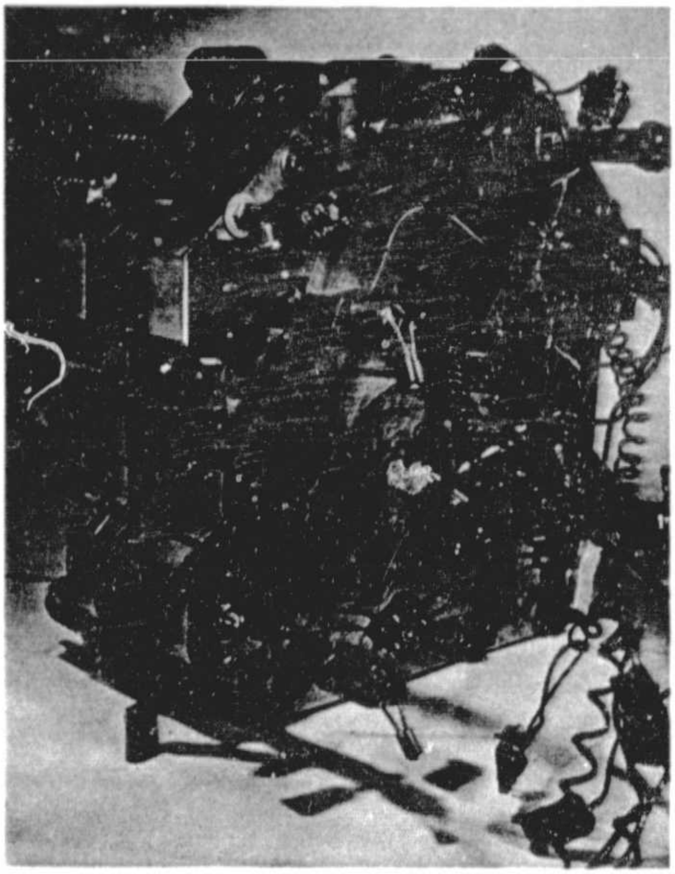

Figure 4.
Multi-Cell Stack Test Set-Up

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SECTION III.A

A. Long-Term Six-Cell Stack Endurance Test Rig 39578-1

The NASA-Lewis Six-Cell Stack Endurance Test Rig 39578-1 was comprised of six, 0.508 ft^2 (471.9 cm^2) active area "Orbiter-type" cells. The stack was endurance tested for 16,800 hours to a continuous cyclical load profile, shown in Figure 5 which simulates a Regenerative Fuel Cell energy storage system operating in low-earth orbit. The endurance test goal was 20,000-hours.

A total of 18,054 hours of testing including 18,000 hours of cyclical operation simulating 12,000 cycles of Regenerative Fuel Cell operation was completed. The test was conducted at 140°F (60°C) coolant inlet temperature and a 60 psia (41.4 N/cm^2) reactant pressure.

The off-load, open circuit, and on-load, 200 ASF (215.3 MA/cm^2) cell voltage remained stable at less than 4.0 microvolts per hour voltage reduction throughout the test-confirming the ability of the alkaline fuel cell to operate in a cyclical mode, representative of a Regenerative Fuel Cell Energy Storage System.

The NASA-Lewis Six-Cell Stack Rig 39578-1 Endurance Test was voluntarily terminated upon completion of 18,054 hours of cyclical operation in order to conduct a teardown inspection of the rig to provide technical information to assist in specifying the cell configuration for the next planned long-term endurance test rig.

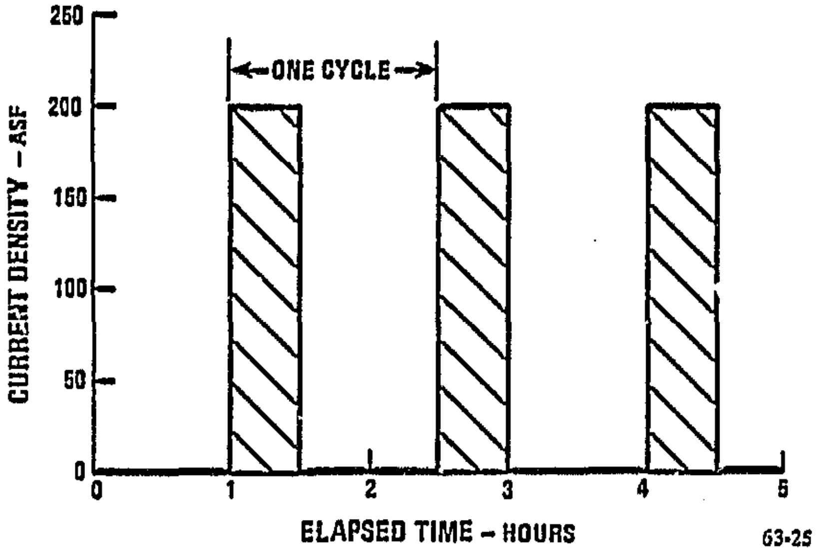

Figure 5. Continuous Cylical Load Profile

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SECTION III.A.1

1. Test Facility Preparation

Refurbishment of test stand X535 was undertaken to improve test stand operating reliability, modernize facilities and upgrade, the test stand to handle long-term endurance testing of multi-cell stacks at steady-state and to cyclical load profiles. The long-term endurance test of NASA-Lewis Six-Cell Stack Rig 39578-1 was conducted in test stand X535.

The NASA-Lewis Cyclical Load Profile Test Facility X535 is shown in Figure 6. Redundant systems including the coolant pump, reactant scrubber, heaters and instrumentation were incorporated into the test stand.

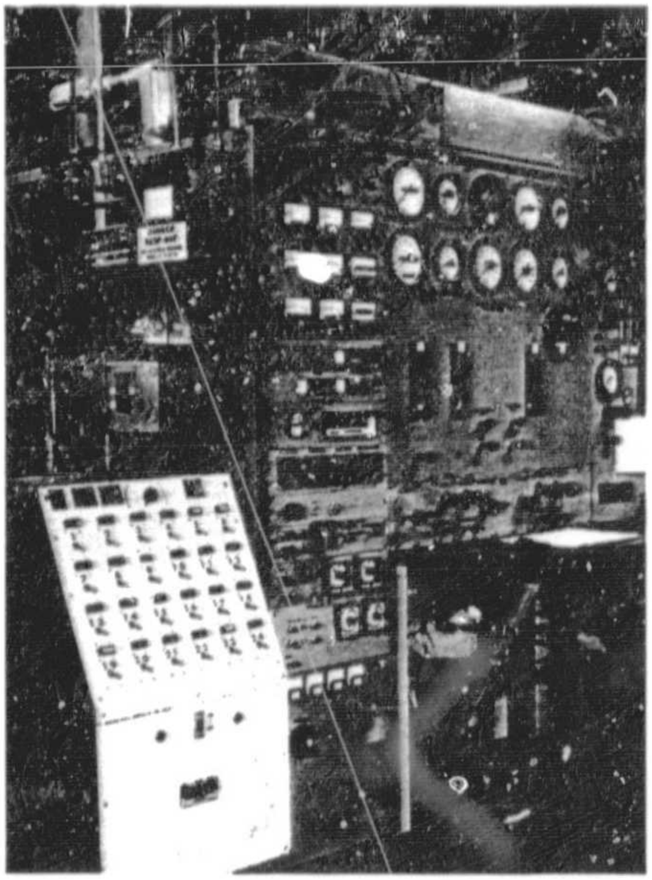

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Figure 6.

NASA-Lewis Cyclical Load Profile Test Facility X535

The Automatic Data Acquisition and Recording (ADAR) system was expanded to incorporate a caution and warning and automatic test stand shutdown when operating parameters or shutdown limits were exceeded. In addition, the ADAR system has back-up channels to monitor shutdown parameters to ensure that the test stand should indeed be shutdown. This approach minimizes the opportunity for instrumentation and or ADAR equipment malfunctions leading to an unnecessary stand shutdown.

A summary of the work completed on X535 in preparation for the long-term cyclical endurance test follows:

- Reactant pressure regulators and stand control valves were cleaned.
- Shut-off valves were installed around critical stand components, such as the coolant pump, hydrogen pump and scrubbers. These valves would permit replacement of these components during the open circuit portion of the operating cycle.
- Increased insulation was installed on the coolant loop to reduce heat loss and improve temperature control.
- Redundant hydrogen and coolant preheaters were installed in the test stand.
- A long-life double-pole load contactor was installed in the test stand.
- Backup temperature controllers were provided in the test stand.

A pump for circulating hydrogen was installed. Recirculating the hydrogen stream more closely will resemble space station operation and reduce electrolyte exposure to CO and CO_2 contamination in the hydrogen. The only hydrogen added to the recirculating system makes up for consumption in the fuel cell stack.

SECTION III.A.2

2. Stack Cell Configuration

The cell configuration for the Long-Term Six-Cell Stack Endurance Test Rig 39578-1 is presented in Table I. Eight cells were fabricated providing two spares for use as replacements during the endurance test. New magnesium separator plates were employed in the assembly of the stack.

TABLE I. CELL CONFIGURATION - RIG 39578-1

• Cathode	
• Catalyst	90 Au-10 Pt
• Loading	20 mg/cm^2 (Nominal)
• Matrix	
• Material	Advanced Reconstituted Asbestos
• Thickness	20-mils (0.51 mm)
• Anode	
• Catalyst	10% Pt Supported on Carbon
• Loading	0.5 mg/cm^2 (Nominal)
• Electrolyte Reservoir Plate	
• Material	Porous Nickel
• Thickness	51 mils (1.3 mm)
• Edge Frame	
• Material	Fiberglass/Epoxy

The stack was filled with 23% wgt KOH by following a laboratory electrolyte fill procedure. This procedure involves flushing the stack with hydrazine diluted electrolyte with a thermal cycle to operating temperature. Following the hydrazine diluted electrolyte flush, the stack is flush refilled with 23% wgt KOH.

Upon completion of the electrolyte fill, rig 39578-1 shown in Figure 7 was delivered to test stand X535 for a performance checkout test and commencement of the planned endurance test.

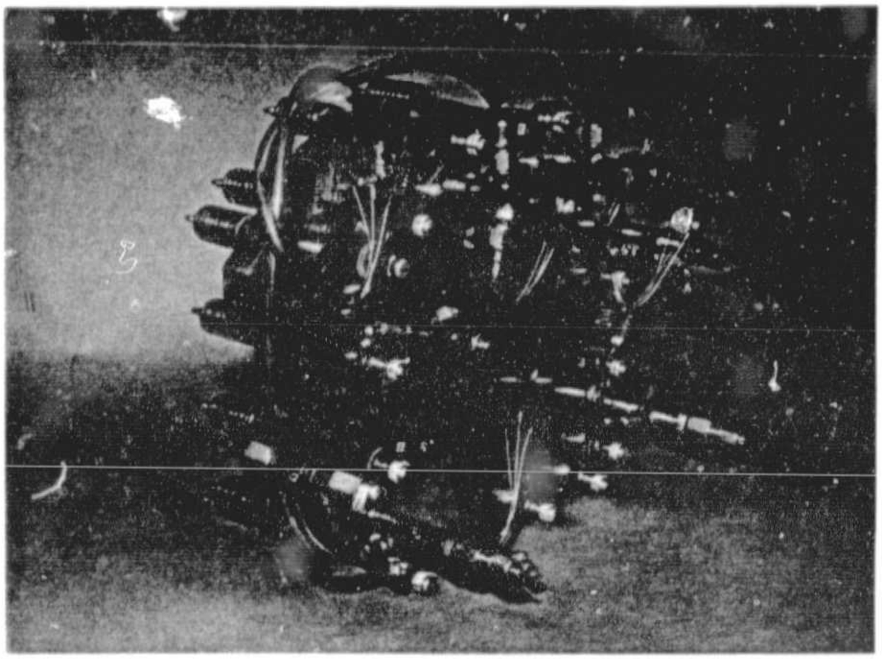

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Figure 7. NASA-Lewis Six-Cell Stack Long-Term Endurance Test Rig 39578-1

SECTION III.A.3

3. Evaluation Test Results

The Long-Term Six-Cell Stack Endurance Test Rig 39578-1 was constructed with six "Orbiter-Type" 0.508 ft^2 (471.9 cm^2) active area cells, featuring platinum-on-carbon catalyst anodes, gold platinum catalyst cathodes and advanced asbestos matrices.

Rig 39578-1 completed a total of 18,054 hours of testing which included 18,000-hours of operation to a cyclical load profile consisting of 60-minutes at open circuit followed by 30-minutes on-load. The 18,000-hours of cyclical operation simulates 12,000-cycles of a Regenerative Fuel Cell Energy Storage System operating in low earth orbit. The endurance test was conducted at a coolant inlet temperature of 140°F (60°C) and a reactant pressure of 60 psia (41.4 N/cm^2).

The performance history of rig 39578-1 for 16,800 hours of testing at 200 $amps/ft^2$ (215.3mA/cm^2) is presented on Figure 8. The average cell performance following 18,054 hours of operation was 0.885 V/C at 100 $amps/ft^2$ (107.6 mA/cm^2). The individual cell performance at 18,054 hours ranged from 0.818 V/C to 0.933 V/C. The average cell open circuit was 1.104 V/C.

The endurance test of rig 39578-1 was voluntarily terminated after 18,054-hours of operation in order to conduct a teardown inspection of the rig to provide technical information to assist in specifying the cell configuration for the next planned long-term endurance test rig.

A teardown inspection plan for rig 39578-1 has been prepared. The teardown will be conducted under the continuation of the Lewis Research Center technology advancement program.

There were only three performance anomalies identified during the endurance test. The anomalies were a reduced open circuit (O/C) voltage on Cell No. 5, apparent port plugging on Cell No. 2 and a low performance incident on Cell No. 6.

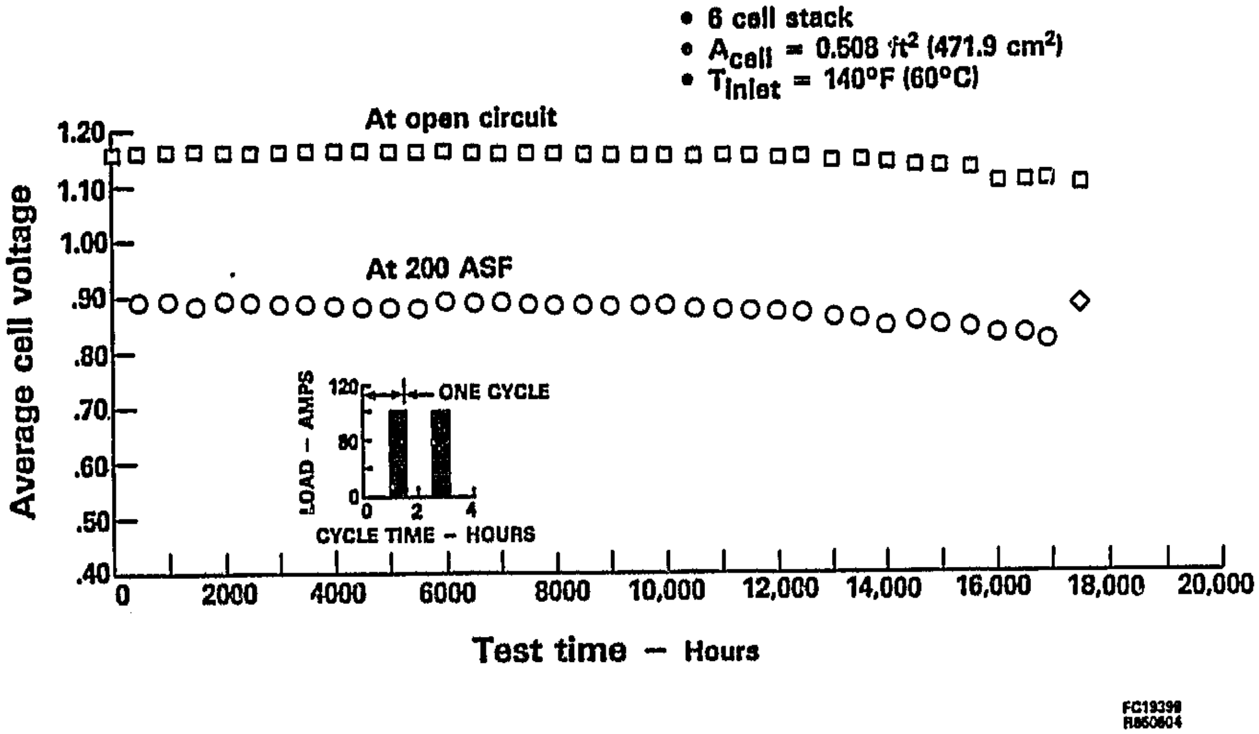

Figure 8. NASA-Lewis Six-Cell Stack Long-Term Endurance Test Performance History (Rig 39578-1)

The O/C voltage of Cell No. 5 experienced a slow but steady decline during the last 10,000-hours of operation. There are two possible explanations for the reduction with time of Cell No. 5 O/C voltage. A reactant gas-to-gas leak or an internal electrical short could explain the anomaly. An internal electrical short was suspected, since there was no evidence of performance instability or temperature increase that would be expected with a gas-to-gas leak. Performance checkout tests conducted prior to shutdown indicated an equivalent short of 1.2 amps/ft² (1.3 mA/cm²) which is considered to be insignificant.

A greater performance recovery following reactant purges on Cell No. 2 was primary evidence of port plugging. Other than a reduced performance level due to inert buildup, the port plugging on Cell No. 2 did not interfere with the continuation of the endurance test.

A sudden low voltage occurred on Cell No. 6 at 16,800-hours. At this point, the voltage on Cell No. 6 could not be maintained at 200 amps/ft^2 (215.3 mA/cm^2). Following the incident, the load was removed and rig 39578-1 was maintained at temperature, at open circuit, overnight. The low voltage could have been the result of port plugging and/or performance sensitivity of the cell to electrolyte concentration induced by variation in stack operating conditions. As a result of the low voltage incident, the cyclical endurance test was completed at an on-load current density of 100 amps/ft^2 (107.6 mA/cm^2).

Data generated during the planned teardown investigation will be available to help in identifying the causes of the endurance test performance anomalies.

SECTION III.B

B. Advanced Technology Four-Cell Stack Rig 39673-1

The Advanced Technology Stack Rig 39673-1 was assembled with four-cells incorporating supported platinum-on-carbon catalyst anodes, butyl bonded potassium titanate matrices, and low weight carbon electrolyte reservoir plates. All cells within the stack are constructed to the same cell configuration.

A total of 2860 hours of testing at an endurance load of 100 ASF (107.6 mA/cm^2), a coolant inlet temperature of 180°F (82.2°C) and a 60 psia (41.4 N/cm^2) reactant pressure has been completed during the program. The endurance test is continuing.

Test results to date verify the suitability of the supported catalyst anode, bonded potassium titanate matrix and carbon electrolyte reservoir plate for use in the alkaline fuel cell. The supported platinum catalyst anode as a result of its performance stability has the potential to extend endurance. The corrosion-resistance bonded potassium titanate matrix should contribute to an extended cell endurance capability. The low weight of the carbon electrolyte reservoir plate would reduce the weight of a standard production cell by nearly one-half.

At 2208 load hours, a low voltage on Cell No. 1 and an increase in the coolant exit temperature led to stopping the endurance test. Pressure testing conducted following cooldown to ambient confirmed reactant cross leakage on Cell Nos. 1 and 4. Inspection of these cells revealed no significant frame discoloration or visual evidence of leakage. These two cells were subsequently replaced and the endurance test of the stack resumed.

The average cell performance following 2,860 hours of testing is 0.952V at 100 ASF. For comparison, the initial average cell performance was 0.956V.

SECTION III.B.1

1. Test Facility Preparation

Refurbishment of Test stand X531 was undertaken to improve test stand operating reliability, modernize facilities and upgrade the test stand to handle long-term endurance testing of multi-cell stacks at steady-state and to cyclical load profiles. The 5000-hour endurance test of NASA-Lewis Four-Cell Stack Rig 39673-1 is being conducted in test stand X531.

The NASA-Lewis Cyclical Load Profile Test Facility X531 is shown in Figure 9.

The refurbishment of test stand X531 was similar to that performed on test stand X535 discussed in Section III.A.1 in which the NASA-Lewis Six-Cell Stack Long-Term Endurance Test Rig 39578-1 was conducted.

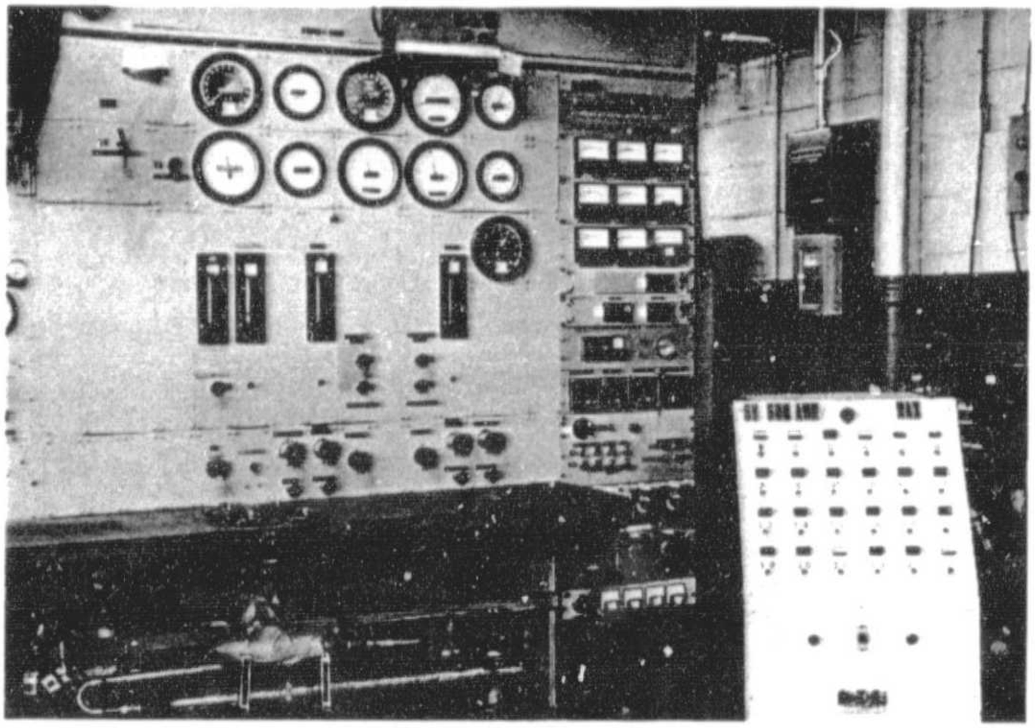

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Figure 9. NASA-Lewis Cyclical Load Profile Test Facility

SECTION III.B.2

2. Stack Cell Configuration

The description of the cell configuration evaluated in the Advanced Technology Four-Cell Rig 39673-1 is presented in Table II. Six cells were fabricated providing two spares for use as replacements during the endurance test. Used magnesium separator plates were employed in the assembly of the stack.

TABLE II. CELL CONFIGURATION - RIG 39673-1

● Cathode	
● Catalyst	90 Au-10 Pt
● Loading	20 mg/cm^2 (Nominal)
● Matrix	
● Material	96% Potassium Titanate 4% Butyl Rubber
● Thickness	20-mils (0.5 mm)
● Anode	
● Catalyst	10% Pt Supported on Carbon
● Loading	0.5 mg/cm^2 (Nominal)
● Electrolyte Reservoir Plate	
● Material	Porous Carbon (Nickel Impregnated)
● Thickness	70 mils (1.8 mm)
● Edge Frame	
● Material	Fiberglass/Epoxy

A photograph of the carbon electrolyte reservoir plate is shown in Figure 10.

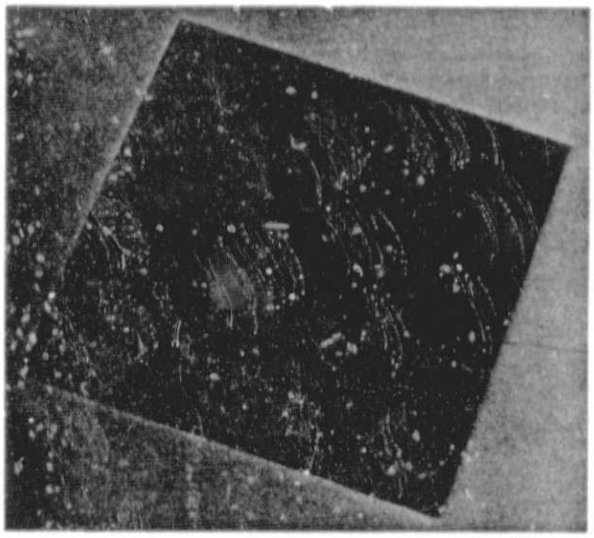

(WCN-11031)

Figure 10. Lightweight Carbon Electrolyte Reservoir Plate

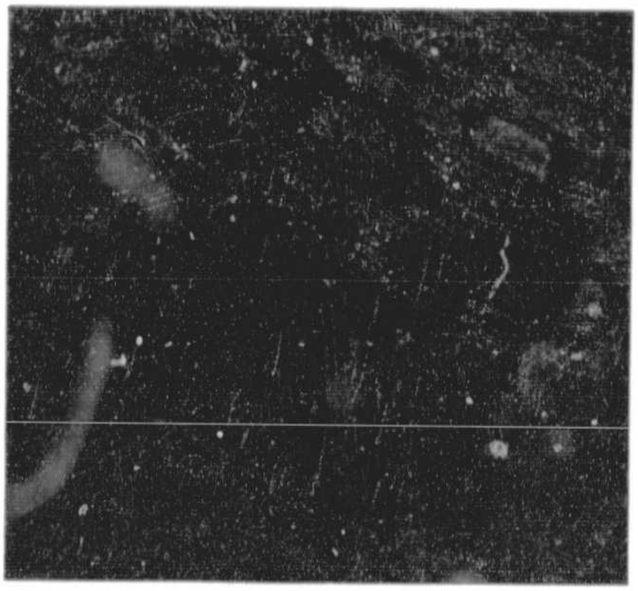
A method for fabricating 0.508 ft^2 ($471.9cm^2$) active area free standing butyl rubber bonded potassium titanate matrices was identified during cell unitizing trials. A total of 50 full-size, mat-type matrices were fabricated during these trials. Repeatable bubble pressure measurements of over 20 psi (13.8 N/cm^2) were obtained on the matrices from the final fabrication lot.

The hydrogen side (anode) and oxygen side (cathode) of an assembled cell is shown on Figure 11. The cathode featured standard "Orbiter-type" foils at the inlets to permit water-vapor back diffusion for inlet humidification.

Two laboratory research cells, incorporating the electrodes and matrix configuration of the four-cell stack, were constructed to identify an electrolyte fill procedure. Test results confirmed that the standard laboratory fill procedure, discussed below, was the best approach.

The stack was filled with 23% wgt potassium hydroxide by following a laboratory electrolyte fill procedure. This procedure involves flushing the stack with hydrazine diluted electrolyte with a thermal cycle to operating temperature. Following the hydrazine diluted electrolyte flush, the stack is flush refilled with 23% wgt. KOH.

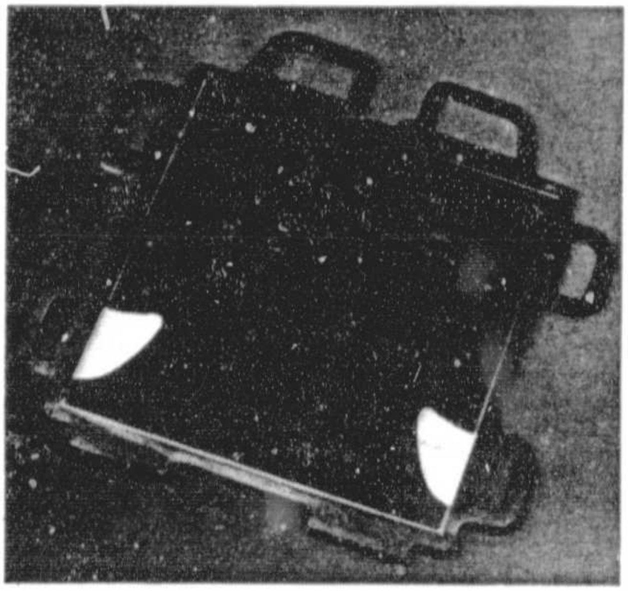
Upon completion of the electrolyte fill, the stack was delivered to test stand X531 for a performance checkout test and commencement of the planned endurance test.



HYDROGEN ELECTRODE

(WCN-11029)

OXYGEN ELECTRODE



(WCN-11033)

Figure 11. Assembled Cell Configuration For Four-Cell Stack Rig 39673-1

SECTION III.B.3

3. Evaluation Test Results

The advanced technology four-cell Rig 39673-1 has completed 2860-hours of a planned 5000-hour endurance test. The test is being conducted at a coolant inlet temperature of 180°F (82.2°C) a 60 psia (41.4 N/cm^2) reactant pressure and a steady-state load of 100 amps/ft^2 (107.6 mA/cm^2).

The performance history of Rig 39673-1 is presented on Figure 12. The initial average cell voltage was 0.956 V at 100 amps/ft^2 (107.6 mA/cm^2) and following 2860-hours of testing average cell voltage is 0.952 V/C. A cell voltage profile over the course of the test is presented on Figure 13. The endurance test is continuing.

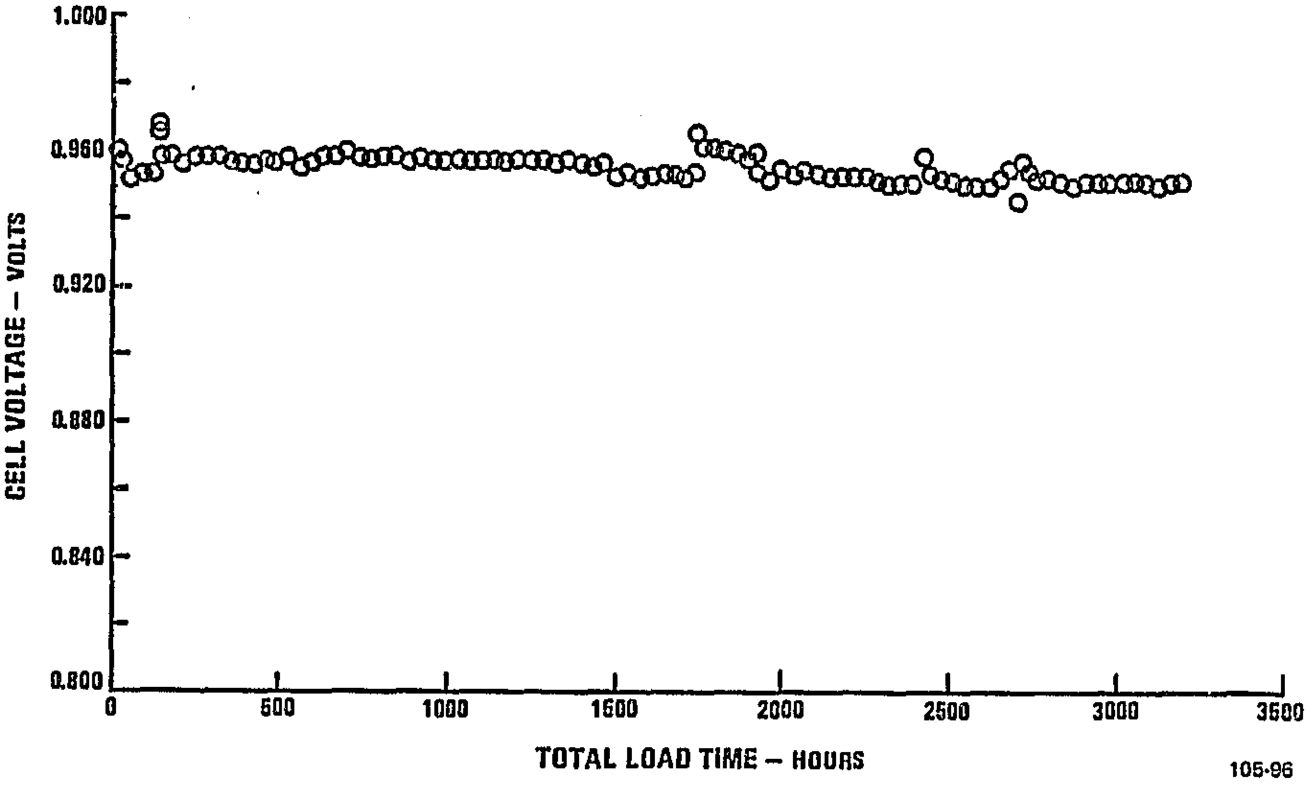
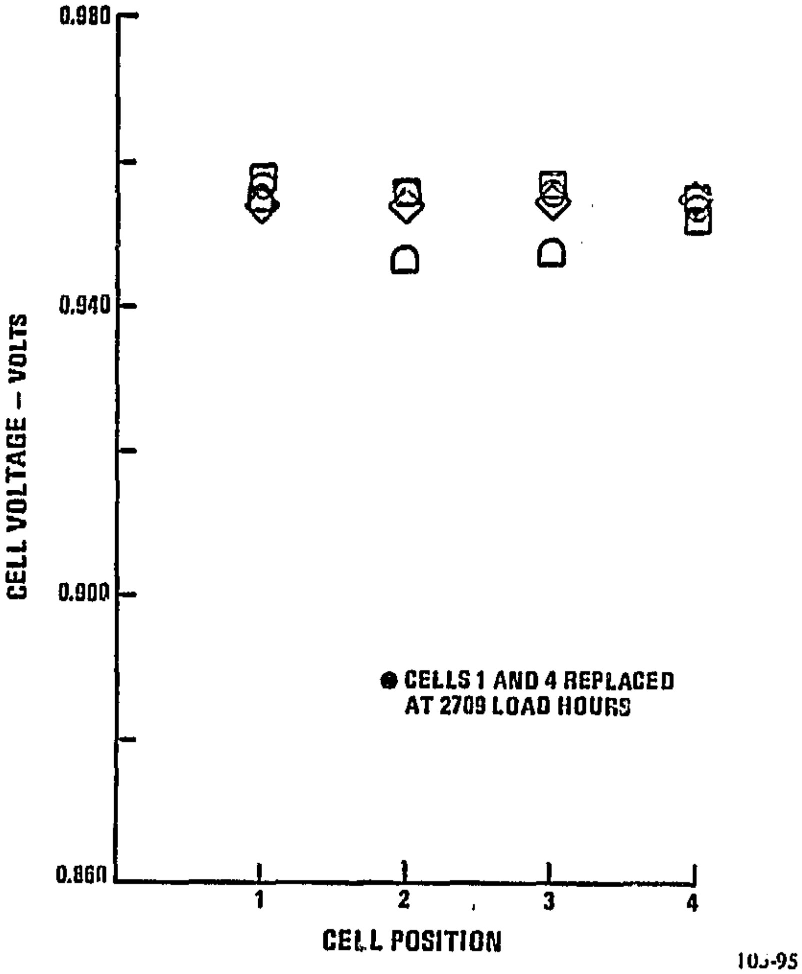


Figure 12. Advanced Technology Four-Cell Stack Rig 39673-1 Performance History

	DATE	TIME	LOAD TIME	AMPS	AVG V/C	COOL IN	COOL OUT	H_2 D.P.
○	6-17-84	834	42	49.84	.956	181	195	167
□	6-27-84	1705	1000	48.40	.956	179	194	168
◇	8-19-84	0106	2031	50.36	.953	178	193	166
∩	10-30-84	1854	2862	49.59	.952	170	193	167



105-95

Figure 13. Advanced Technology Four-Cell Stack Cell Voltage Profile (Rig 39673-1)

At 2208 load hours, a low voltage on Cell No. 1 and an increase in coolant exit temperature led to stopping the endurance test. Internal reactant cross-leakage was suspected. Pressure testing conducted following cooldown to ambient confirmed reactant cross-leakage on Cell Nos. 1 and 4. Inspection of these cells revealed no significant frame discoloration or visual evidence of leakage.

Two spare cells from the program containing bonded potassium titanate matrices and graphite electrolyte reservoir plates were used as replacements in the four-cell stack. Following completion of an electrolyte fill of the replacement cells and stack reassembly, the endurance test was resumed.

The test objectives are to demonstrate the potential of the supported catalyst anode and the corrosion resistant butyl bonded potassium titanate matrix to extend cell endurance capability. In addition the suitability of the carbon electrolyte reservoir plate which would reduce the weight of the standard production cell by nearly fifty percent is being demonstrated.

SECTION III.C

C. ADVANCED TECHNOLOGY FOUR-CELL STACK RIG 39493-1

The Advanced Technology Stack Rig 39493-1 was assembled with four-cells incorporating supported platinum-on-carbon anodes, advanced asbestos and asbestos reinforced potassium titanate matrices, and low weight carbon electrolyte reservoir plates.

A total of 3,500-hours of testing at an endurance load of 100ASF (107.6 mA/cm^2), a coolant inlet temperature of 180°F (82.2°C) and a 60 psia (41.4 N/cm^2) reactant pressure was completed.

The performance stability of the supported platinum catalyst anodes demonstrated their potential for extended endurance. Testing has also shown the low weight carbon electrolyte reservoir plate is suitable for use in the alkaline fuel cell.

SECTION III.C.1

1. Stack Cell Configuration

The description of the individual cell configuration evaluated in Advanced Technology Four-Cell rig 39493-1 is presented in Table III.

Cell numbers 1 and 4 were constructed with advanced asbestos matrices and hybrid polysulfone cell edge frames. The advanced asbestos matrix configuration was developed by Power Systems Division under the Orbiter Fuel Cell Contract with Rockwell International. The hybrid polysulfone frame was developed under a Lewis Research Center program (reference 7) for a small passive water removal fuel cell approach. Cell numbers 2 and 3 were constructed with matrices composed of 80% potassium titanate reinforced with 20% asbestos.

TABLE III. ADVANCED TECHNOLOGY FOUR-CELL STACK RIG 39493-1

Cell No.	Anode Catalyst	Cathode Catalyst	Matrix	Edge Frame	Electrolyte Reservoir Plate
1	Pt/C	AuPt	Asbestos	Hybrid Polysulfone	Carbon
2	Pt/Pd	AuPt	Potassium Titanate Asbestos	Epoxy/Fiberglass	Nickel
3	Pt/C	AuPt	Potassium Titanate Asbestos	Epoxy/Fiberglass	Carbon
4	Pt/C	AuPt	Asbestos	Hybrid Polysulfone	Nickel

SECTION III.C.2

2. Evaluation Test Results

The second advanced technology four-cell stack rig 39493-1 completed 3,500 hours of operation at a coolant inlet temperature of 180°F (82.2°C), a 60 psia (41.4 N/cm^2) reactant pressure and a current density of 100 ASF (107.6 mA/cm^2). The endurance test was conducted under NASA Contract No. NAS3-21293 (Reference 4).

Reactant cross-pressure tests, conducted prior to the planned resumption of the endurance test following a four-month shutdown due to a lack of a contract, revealed the presence of reactant cross-leakage within the stack. This leakage precluded further endurance testing.

The performance history of rig 39493-1 is presented in Figure 14.

The stack incorporates cells featuring platinum-on-carbon catalyst anodes, gold-platinum catalyst cathodes, asbestos reinforced potassium titanate matrices and carbon electrolyte reservoir plates. A detailed description of the four-cell stack construction can be found in Table III, Section III.

A teardown inspection of the stack and individual cells was conducted under the program. The following discussion summarizes the findings of the inspection.

The exterior of the stack appeared to be in good condition. There were scattered small white deposits, probably potassium carbonate, at the junction of the cell and magnesium separator plate. These deposits could result from the carbonation of potassium hydroxide residue left in these areas following cell electrolyte filling.

Pressure testing of the stack revealed no reactant or coolant to external or reactant-to-coolant leakage at the stack operating pressure of 60 psia (41.4 mA/cm^2). As expected, internal reactant leakage was observed.

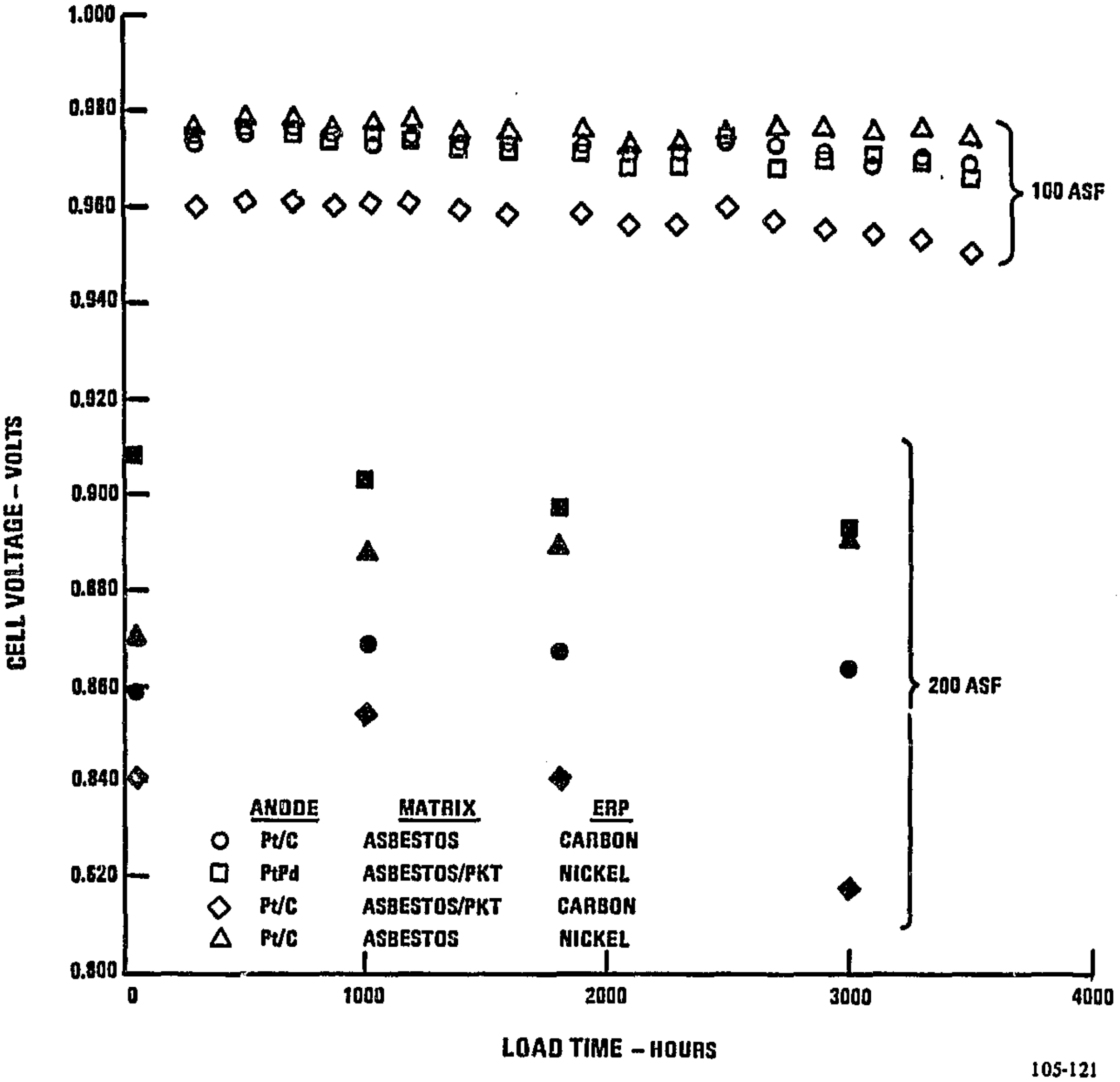


Figure 14. Second Advanced Technology Four-Cell Stack Rig 39493-1 Performance History

The next step in the teardown was to disassemble the stack. Visual inspection of the cell edge frames found them to be in good condition. There was some evidence of discoloration and leaching of the epoxy from the frame especially on the cathode side. The leaching and discoloration of the frames has been observed on previous cells which had been endurance tested and is considered normal.

The individual cells were mounted between plexiglass end-plates for reactant cross pressure testing. Testing revealed that Cell No. 4 had an internal reactant leak. Cell No. 4 had been identified prior to teardown as the most likely cell to be experiencing internal reactant leakage. This cell prior to stopping the stack endurance test had experienced a reduction in open-circuit voltage and a depression in Tafel performance; both indicators of an electrical short or reactant cross leakage.

Inspection of the gold-plated magnesium separator plates found them to be in good condition. All the metering ports, oxygen, hydrogen, and coolant were open and clean.

A summary of the results from the inspection of individual cells is presented in the following discussions.

<u>Cell No. 1</u>

- There were indentations observed in the carbon reservoir plate (ERP) from the adjoining separator plate. These indentations, which did not affect cell performance, more than likely resulted from the use of available "soft" carbon ribbed substrate (ERP) and the assembly of the cells between existing separator plates.

- There was a slight discoloration along the bottom of the ERP.

Cell No. 2

- Evidence of potassium carbonate was observed around the O_2 inlet foil. The highest electrolyte concentration, occurs at the O_2 inlet. The purpose of the foils at the O_2 inlet is to prevent localized dryout. However, precipitation of potassium carbonate around the periphery of the foil is not unusual due to reduced water-vapor pressure (elevated electrolyte concentration) in this location.

Cell No. 3

- Indentations in the carbon ERP from the adjacent separator plate were observed. These indentations were similar to those found in Cell No. 1.
- Potassium carbonate deposits were noted around the O_2 inlet foil.

Cell No. 4

- The cathode separated from the hybrid polysulfone frame in the area of the hydrogen exit and coolant exit manifolds.
- Cell edge frame discolored in the area where the cathode had separated from the frame.
- Crosspressure test confirmed this cell to have reactant cross leakage.

Cell No. 1 (Hybrid polysulfone frame) and Cell No. 2 (Fiberglass/epoxy frame) were submitted to the laboratory for a carbonate analysis of the cell electrolyte. Table IV presents the results of the carbonate analysis.

TABLE IV. SECOND ADVANCED-TECHNOLOGY FOUR-CELL STACK CARBONATE ANALYSIS

	Edge Frame	ERP	% Conversion of KOH to K_2CO_3
Cell No. 1	Hybrid Polysulfone	Carbon	12.0
Cell No. 2	Fiberglass/Epoxy	Nickel	20.8

The results of the carbonate analysis of cell No. 2 were consistent with past test results of fiberglass/epoxy frame cells. The 42 percent lower carbonate level of Cell No. 1 with the hybrid frame is consistent with the results from the hybrid frame cell in the First Advanced-Technology Stack, Rig 39461-1, reported in NASA CR-165417. In addition the carbonate results verify that the carbon ERP and the platinum-on-carbon catalyst anode are stable and do not contribute significantly to carbonation of the electrolyte.

Cell No. 4 was mounted in a clear plexiglass test fixture to identify the site of the internal reactant leak. The reactant side of the cathode was filled with water and the anode was pressurized with nitrogen. The leakage site was identified to be at the electrode edge of the cell frame in the middle of the coolant-exit manifold by observing nitrogen bubbles in the water at the site.

Reactant cross leakage occurred as a result of insufficient encapsulation of the electrodes and matrix by the hybrid polysulfone frame. The frame construction employs numerous thin polysulfone "strips" for the encapsulation and sealing which are difficult to maintain in alignment during cell unitizing.

IV. FUEL CELL MATRIX RESEARCH

An experimental program was conducted to identify an alternate matrix configuration. The objective of the work was to identify a matrix configuration capable of operating on hydrogen and oxygen at a 60 psia (41.4 N/cm^2) reactant pressure at a cell temperature of 250°F (121.1°C).

The primary approach was to fabricate and evaluate fuel cell matrices comprised of fine particulates bonded with organic resins. A secondary activity was to evaluate the suitability of microporous membranes for service in hydrogen-oxygen alkaline electrolyte fuel cells.

The candidate matrix particulate and binder materials that were evaluated under the program are identified in Table V.

Table V. Candidate Matrix Particulate And Binder Materials

- Candidate Particulate Materials
  - Potassium Titanate (PKT)
  - Silicon Carbide (SiC)
  - Zirconium Oxide (ZrO_2)
- Candidate Binder Materials
  - Butyl Rubber
  - Polysulfone
  - Polyphenylsulfone (PPS)
  - Polyethersulfone (PES)
  - Teflon®

Corrosion tests in hot aqueous potassium hydroxide were conducted to identify alternate corrosion-resistant particulate materials. Matrices were fabricated from these materials by bonding them with butyl rubber or Teflon®. These matrices were incorporated into laboratory research cells and performance evaluated.

The particulate materials corrosion tested in 42% wgt potassium hydroxide of 250°F (121.1°C) are presented in Table VI.

Table VI. Advanced Matrix Particulate Materials

- Polyetherimide
- Ceria, CeO_2
- Zirconia
- Lithium Aluminate, $LiAlO_2$
- Strontium Titanate, $SrTiO_3$
- Lithium Titanate, Li_2TiO_3
- Strontium Zirconate, $SrZrO_3$
- Lithium Stannate, Li_2SnO_3
- Lithium Cobaltate, $LiCoO_2$
- Lanthanum Cobaltate, $LaCoO_3$
- Lanthanum Manganate, $LaMnO_3$
- Magnesium Zirconate, $MgZrO_3$

An exploratory evaluation of bonded polyantimonic acid and fibrous polybenzimidazole matrices was conducted. The evaluation of these corrosion-resistant materials consisted of fabrication trials to develop a matrix structure culminating in research cell endurance testing of cells incorporating the matrix.

An advanced composite matrix consisting of a potassium titanate layer deposited onto an advanced asbestos layer was evaluated. The corrosion resistance of the potassium titanate layer in the composite matrix reduces electrolyte contamination thereby improving the long-term performance characteristics of the alkaline fuel cell.

A. PARTICULATE MATERIAL CORROSION TESTS

The particulate and binder materials identified in Table V have demonstrated corrosion-resistance in hot aqueous potassium hydroxide. The early fabrication trials conducted under the program culminated in the successful development of a free-standing butyl rubber bonded potassium titanate matrix with a bubble pressure greater than 20 psi (13.8 N/cm²).

Corrosion testing of the particulates identified in Table VII was conducted to identify alternate particulate matrix materials. The materials were corrosion tested for 3000-hours to the procedure outlined in Section IV.H.1.. Periodically during the corrosion test, samples were subjected to Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) analysis. Corrosion test results are summarized in Table VII.

Table VII. Particulate Material Corrosion Test Results

Material	Time (Hrs)	Crystal Phase (By XRD)	Grain Size (By SEM)
CeO_2	0	100% f.c.c. CeO_2	
	250	No Change	No Change
	500	No Change	No Change
	1000	No Change	No Change
	3000	No Change	No Change
ZrO_2	0	80% Monoclinic ZrO_2; 20% Cubic ZrO_2	
	250	75% Monoclinic ZrO_2; 25% Cubic ZrO_2	No Change
	500	80% Monoclinic ZrO_2; 20% Cubic ZrO_2	No Change
	1000	80% Monoclinic ZrO_2; 20% Cubic ZrO_2	No Change
	3000	80% Monoclinic ZrO_2; 20% Cubic ZrO_2	No Change
$LiAlO_2$	0	95-97% Tetragonal γ-$LiAlO_2$ 2-1% -γ-Al_2O_3 2-1% $(K, Li)_2\ Al_2O_4 - yH_2O$ 1% $LiAl_5O_8$	

Table VII. Particulate Material Corrosion Test Results (Cont'd)

Material	Time (Hrs)	Crystal Phase (By XRD)	Grain Size (By SEM)
$LiAlO_2$	250	50% Tetragonal γ-$LiAlO_2$ 45% $(Li, K)_2 Al_2O_4 \cdot yH_2O$ 5% Hexagonal $\kappa AlSiO_4$	Agglomeration and Grain Growth
	500	50% $(Li, K)_2 Al_2O_4 \cdot yH_2O$ 40% Tetragonal γ-$LiAlO_2$ 10% Hexagonal $\kappa AlSiO_4$	Continued Grain Growth
	1000	100% Hexagonal $\kappa AlSiO_4$	Smoothing of Agglomerated Grains
	3000	90% Hexagonal $\kappa AlSiO_4$ 10% Hexagonal γ-$(Li, K)_2 Al_2O_4 \cdot yH_2O$	No Further Change
$SrTiO_3$	0	100% Cubic $SrTiO_3$	
	250	No Change	No Change
	500	No Change	No Change
	1000	No Change	No Change
	3000	No Change	No Change
Li_2TiO_3	0	95% Monoclinic Li_2TiO_3 5% Li_4GeO_4 Type Structure	
	250	No Change	Agglomeration
	500	No Change	Grain Growth
	1000	No Change	Smoothing of Agglomerates
	3000	90% Monoclinic Li_2TiO_3 10% Li_4GeO_4 Type Structure	No Further Change

Table VII. Particulate Material Corrosion Test Results (Cont'd)

Material	Time (Hrs)	Crystal Phase (By XRD)	Grain Size (By SEM)
$SrZrO_3$	0	100% Orthorhombic $SrZrO_3$	
	250	95% Orthorhombic $SrZrO_3$	Grain Growth
		5% Contaminant	
	500	100% Orthorhombic $SrZrO_3$	Grain Growth
	1000	98% Orthorhombic $SrZrO_3$	Same
		2% Contaminant	
	3000	100% Orthorhombic $SrZrO_3$	Same
Li_2SnO_3	0	95% Hexagonal Li_2SnO_3	
		5% Tetragonal SnO	
	250	95% Hexagonal Li_2SnO_3	N.A.
		5% Tetragonal SnO	
	500	Dissolved	N.A.
	1000	Dissolved	N.A.
	3000	Dissolved	N.A.
$LiCoO_3$	0	95% Hexagonal $LiCoO_3$	Agglomerates
		5% f.c.c. Co_3O_4	
	250	No Change	No Change
	500	No Change	No Change
	1000	100% Hexagonal $LiCoO_3$	No Change
	3000	95% Hexagonal $LiCoO_3$	
		5% f.c.c. Co_3O_4	No Change
$LaCoO_3$	0	95% Hexagonal $LaCoO_3$	
		5% Hexagonal $La(OH)_3$	
	250	60% Hexagonal $La(OH)_3$	Grain Growth and
		30% Hexagonal $LaCoO_3$	Needle Formation
		5% f.c.c. Co_3O_4	
	500	Same as 250 Hours	Same
	1000	90% Hexagonal $La(OH)_3$	Grain Growth
	3000	10% Hexagonal $LaCoO_3$	Smaller, Rod-Shaped
		No Sample	Particles

Table VII. Particulate Material Corrosion Test Results (Cont'd)

Material	Time (Hrs)	Crystal Phase (By XRD)	Grain Size (By SEM)
$LaMnO_3$	0	75% Hexagonal $LaMnO_3$	
		25% Hexagonal $La(OH)_3$	
	250	70% Hexagonal $LaMnO_3$	Grain Growth
		30% Hexagonal $La(OH)_3$	
	500	75% Hexagonal $LaMnO_3$	Grain Growth and Smoothing
		25% Hexagonal $La(OH)_3$	
	1000	55% Hexagonal $LaMnO_3$	Agglomeration
		45% Hexagonal $La(OH)_3$	
	3000	55% Hexagonal $LaMnO_3$	
		35% Hexagonal $La(OH)_3$	Same
		10% Orthorhombic $LiLaO_2$	
$MgZrO_3$ ($MgO-ZrO_2$)	0	75 v/o fcc ZrO_2	
		15 v/o Monoclinic ZrO_2	
		10 v/o fcc MgO	
	250	85 v/o fcc ZrO_2	Slight Grain Growth
		10 v/o Monoclinic ZrO_2	
		5 v/o fcc $Mg(OH)_2$	
	500	85 v/o fcc ZrO_2	Same
		10 v/o Monoclinic ZrO_2	
		5 v/o fcc $Mg(OH)_2$	
	1000	80 v/o fcc ZrO_2	Rod Formation, Agglomeration
		10 v/o Monoclinic ZrO_2	
		10 v/o fcc $Mg(OH)_2$	
	3000	80 v/o fcc ZrO_2	Grain Growth, Agglomeration
		10 v/o Monoclinic ZrO_2	
		10 v/o fcc $Mg(OH)_2$	

Samples of polyethermide completely dissolved in 250-hours exposure at 250°F (121.1°C) to 42% wgt. potassium hydroxide.

Corrosion test results presented in Table VII have revealed five particulate materials as corrosion-resistant since there was no appreciable change in crystal structure after 3000-hours immersion in 42% wgt. potassium hydroxide at 250°F (121.1°C). These materials are Ceria, Zirconia, Strontium Titanate, Strontium Zirconate and Lithium Cobaltate.

Employing Teflon® or butyl rubber as a binder, bonded particulate matrices were fabricated from each of the corrosion resistant particulates.

Three polytriazole samples and a triazole modified styrene-isoprene copolymer received from the University of Florida were corrosion tested in aqueous potassium hydroxide. These materials were tested for 100-hours at 250°F (121.1°C) in 42% wgt. KOH. Post-test examination revealed that the three polytriazole samples completely disappeared. The copolymer sample lost 2.5% in weight and turned brown. Test results indicate that these materials are unsuitable for use in the alkaline fuel cell.

B. BONDED PARTICULATE MATRIX FABRICATION

The size range of matrix particulate materials required to achieve acceptable bubble pressure (BP) is a function of particle size and mean pore size (MPS). Work completed under Reference NASA CR-134818 by United Technologies showed that the bubble pressure is related to the mean pore size of a uniform, completely wettable matrix by the relationship:

$$BP(\mathrm{psi}) \sim \frac{42}{MPS\ (\mu)}$$

This relationship assumes densely packed, uniform and spherical particles. As an example to achieve a 45 psi (31.0 N/cm^2) bubble pressure, the fabricated matrix should have a mean pore size of approximately one micron (μ) or less. Therefore where possible, particulate materials employed under the program were in the one micron size range.

Utilizing the particulate and binder materials identified in Table V, four matrix fabrication techniques were evaluated. The techniques included direct filtration, curtain coating, screen printing and spray coating.

Matrices fabricated by direct filtration were the most uniform in structure with the highest repeatable bubble pressure. This method involves filtering a matrix slurry directly onto the electrode substrate or onto a filter paper substrate to produce a handleable, free-standing matrix mat.

Curtain coating and slipcasting were evaluated as techniques to fabricate matrices. A laboratory scale curtain coater was employed initially; however, it was found to have insufficient capacity to produce uniform matrices. Slip casting with a Doctor blade produced uniform matrices with thickness up to 15 mils. Matrices were fabricated from inks formulated from PTFE/PKT, PTFE/SiC, PPS/PKT, PPS/ZrO_2, and Butyl rubber/PKT. However, the maximum bubble pressure achieved using this technique was 15 psi (10.3 N/cm^2).

Screen printed matrix samples prepared by employing a thickening agent yielded thicknesses of only 3-5 mils (.08-.13 mm) in contrast to a desired thickness of 10 to 20 mils (0.5mm).

The spray coating technique of matrix fabrication was ruled out as a viable technique because of poor mixing, uneven structure, and difficulty in depositing the material onto a standard alkaline fuel cell electrode substrate.

The laboratory fabrication trials revealed that matrices fabricated from the particulate potassium titanate and the binders Teflon® and butyl rubber consistently demonstrated the most uniform structures. Work completed under the program focused upon improving the bubble pressure of bonded potassium titanate matrices.

Typical properties of the potassium titanate fibers employed in the matrix work are presented in Table VIII.

Table VIII. Potassium Titanate Fibers
Typical Properties

●	Color	White
●	Bulk Density	0.1 - 0.2
●	Specific Gravity	3.1 - 3.5
●	Melting Point	1250 - 1310°C
●	Tensile Strength	>700 kg/mm^2
●	Specific Heat	0.22
●	Specific Surface Area	1.5 - 2 m^2/g
●	Electric Resistance	3.3 x 10^{15}cmΩ
●	Hardness	4
●	Water Content	<0.7%
●	Odor	None

A scanning electron microscope (SEM) photograph was taken of the potassium titanate fibers manufactured by Otsuka Chemical Co.. A SEM photograph of the fiber is shown in Figure 15. The average fiber diameter of the Otsuka potassium titanate is 0.4μ with a length to diameter of 20 to 1. This data indicates that this material as a non-woven mat should be able to sustain a bubble pressure of 45 psi (31.0 N/cm^2).

Typical properties of the Butyl Rubber (Butyl Latex, BL-100) employed as a binder are presented in Table IX.

Table IX. Butyl Rubber (Butyl Latex BL-100)
Typical Properties

●	Color	White
●	Total Solids Wt. %	62% (range 61-63)
●	Specific Gravity	0.95
●	Density lbs./gallon	7.9
●	pH	5.5 (range 4.5-6.5)
●	Particle Size, Microns	<1 (range 0.1-0.8)
●	Surface Tension at 70°F, dynes/cm	38
●	Viscosity, Brookfield LVT #3 Spindle at 12 RPM	2500 (range 1800-5000)
●	Film tensile strength, psi uncured	35
●	Odor	None

The long-term corrosion resistance of butyl rubber was evaluated. Test samples of pure butyl rubber film showed no significant deterioration in a 5000-hour corrosion test at 250°F (121.1°C) in 42% wgt. potassium hydroxide. However, the test samples became appreciably more tacky between 3000 and 5000 hours.

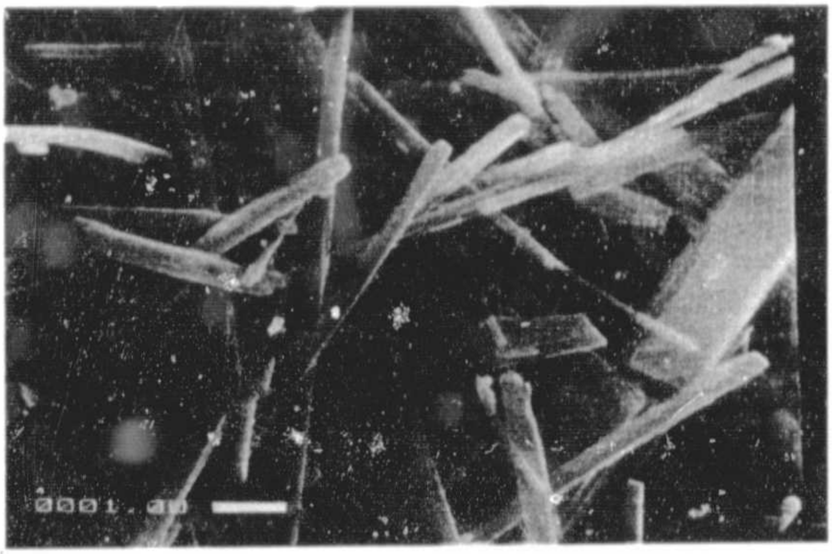


Figure 15. SEM Photograph of Potassium Titanate

A 10-mil (.25mm) thick 95% potassium titanate 5% Teflon® matrix was filtered onto a standard platinum-palladium anode and gold-platinum cathode for evaluation in a laboratory research cell.

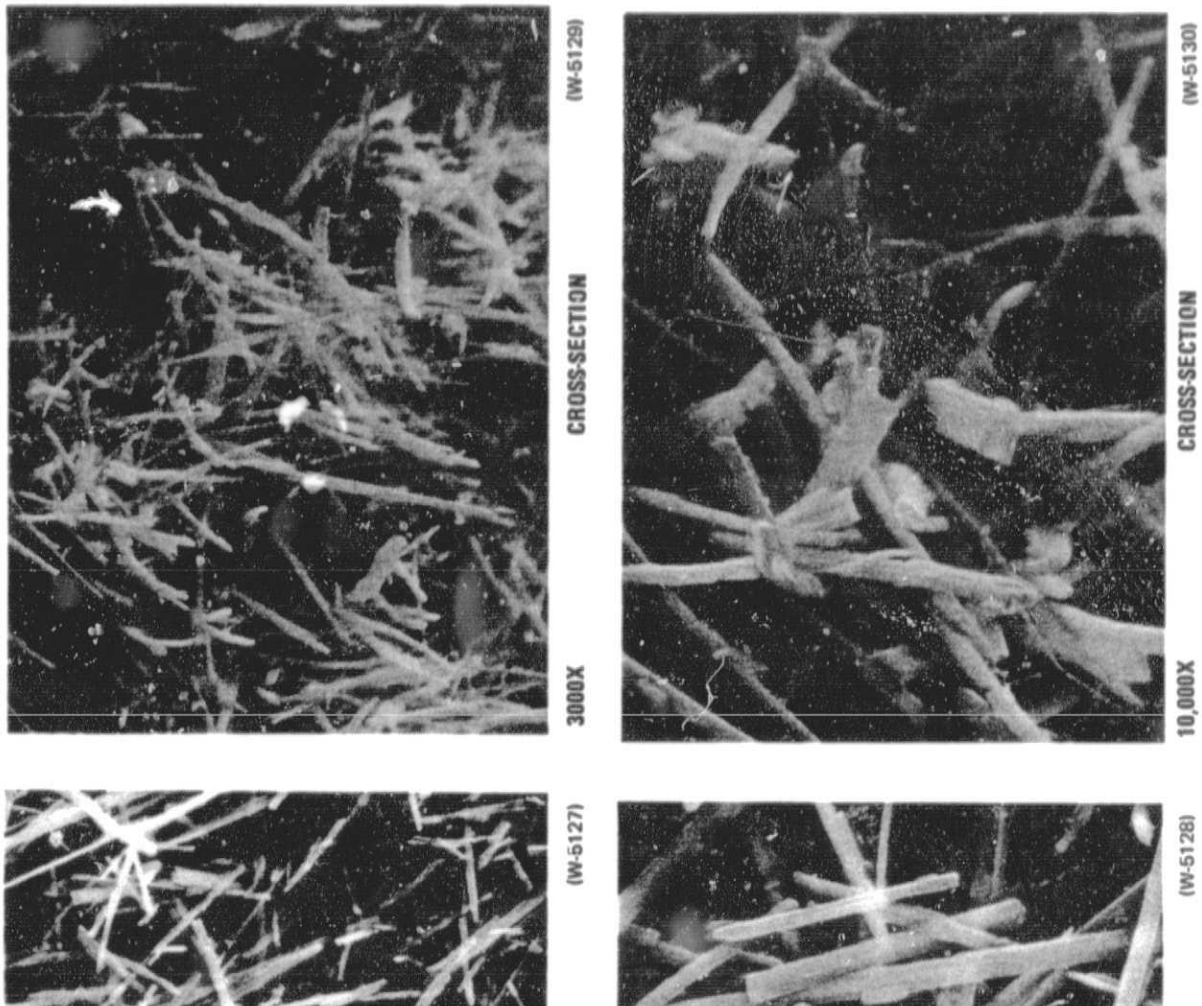
Matrices comprised of 96% potassium titanate and 4% butyl rubber were prepared for evaluation in laboratory research cell endurance tests.

The matrix coating consisting of potassium titanate and butyl rubber were mixed with water and the resultant slurry was filtered directly onto each of the electrodes. The matrix on the anode was 10 mils (.25mm) thick, 70% porous and had a 32 psi (22 N/cm^2) bubble pressure when filled with water. The matrix on the cathode was 10-mils (.25mm) thick, 70% porous, and exhibited a bubble pressure in excess of 50 psi. Standard asbestos matrices have bubble pressures in excess of 50 psi (34.5 N/cm^2). The cathode catalyst layer is thicker and more uniform with smaller holes in the layer compared to the anode catalyst layer, which accounts for the difference in bubble pressure.

In regards to the handleability required for full-size cell unitizing, the butyl rubber bonded matrix is more resilient, scrape-resistant, and rub-resistant than the Teflon® bonded potassium titanate matrix. The butyl bonded matrix was preferred for incorporation into 0.508 ft^2 (471.9 cm^2) active area cell unitizing trials.

Scanning electron micrographs of potassium titanate matrices show that the Teflon® and butyl rubber binders are uniformly distributed throughout the structure. Micrographs of a 95% potassium titanate/5% Teflon® matrix indicated uniformity of the binder, as the presence of the binder is not readily apparent in Figure 16.

The uniform distribution of binder is also seen in the micrographs of a 92% potassium titanate/8% butyl rubber matrix shown in Figure 16. Laboratory size matrices comprised of 96% potassium titanate/4% butyl rubber exhibited a bubble pressure in excess of 50 psid (34.5 N/cm^2).



3000X CROSS-SECTION (W-5129)

10,000X CROSS-SECTION (W-5130)

3000X SURFACE (W-5127)

10,000X SURFACE (W-5128)

Figure 16. Scanning Electron Micrographs (95% - 5% TFE Matrix)

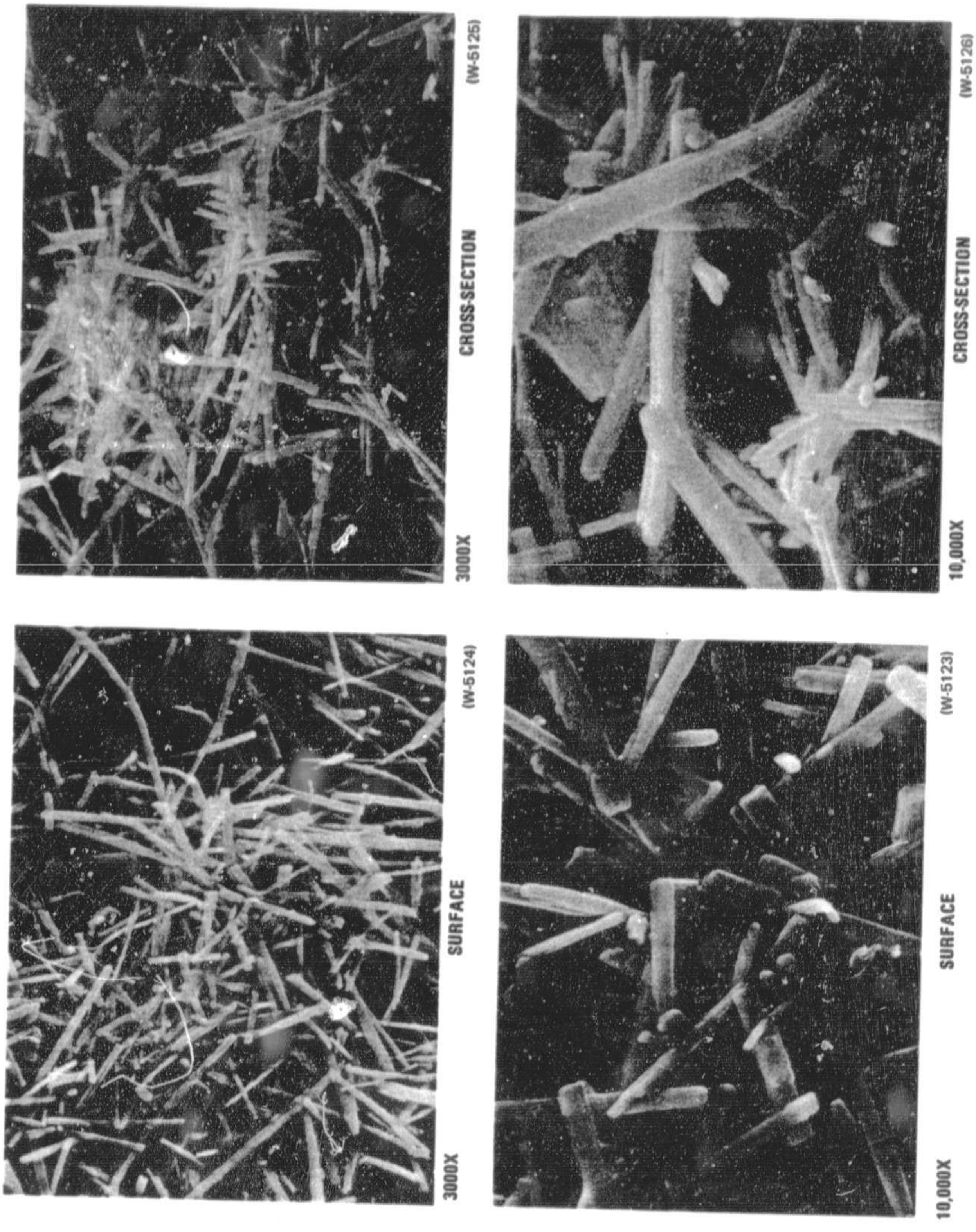


Figure 16. Scanning Electron Micrographs (92% PKT - 8% Butyl Rubber)

Four particulate materials identified as corrosion resistant under Section IV.A were formulated into matrices by bonding with Teflon® or butyl rubber. Bonded strontium titanate, ceria, strontium zirconate and lithium cobaltate matrices were fabricated by a technique which deposits a water dispersion of ceramic particulate and binder onto an electrode substrate. The formulation of the matrices and the research cell that each was incorporated into are identified in Table X.

Table X. Advanced Matrix Configurations

No.	Particulate	Binder	Research Cell No.
1	96% $SrTiO_3$	4% Butyl Rubber	RC-77
2	96% CeO_2	4% Butyl Rubber	RC-76
3	96% $LiCoO_3$	4% Butyl Rubber	RC-92

Strontium zirconate was found to reject butyl rubber in the slurry and segregated during filter deposition. Strontium zirconate bonded with Teflon® was found to be brittle and prone to cracking. Therefore further work with $SrZrO_3$ was stopped.

Each matrix configuration identified in Table X was deposited onto a standard gold-platinum catalyst cathode and platinum-palladium catalyst anode. Approximately 10-mils (.25mm) of the matrix was deposited onto both the anode and cathode, for a total matrix thickness of 20-mils (5mm). A discussion of the test results from the research cell endurance tests is presented in Section V.

C. MEMBRANE EVALUATION

The approach taken in the evaluation of membranes was to identify a commercial membrane which is known to be compatible with the aqueous potassium hydroxide environment.

Two microporous membranes, a fluorocarbon and a polysulfone-based material showed high initial bubble pressure, at greater than 64 psi (44 N/cm²) bubble pressure and water take-up values similar to the asbestos matrix. Table XI presents a summary of the membrane bubble pressure and water-takeup measurements.

Table XI. Membrane Bubble Pressure Results

Membrane	Thickness mils (mm)	Bubble Pressure psi (N/cm²)	Water Takeup % wgt	Remarks
Teflon®	6(.2)	-	4	Leaked
Fluorocarbon Based (FP-W)	4(.1)	>64(44)	50	
Polysulfone Based (HT-100)	5(.1)	>64(44)	60	

Corrosion testing of the FP-W and HT-100 membranes at 250°F (121.1°C) in 42% wgt potassium hydroxide revealed that both membranes became brittle and broke into small fragments. The fluorocarbon based membrane became brittle after only 48-hours and the polysulfone-based membrane by 200-hours. The membrane supplier suggested that the embrittlement problem resulted from the loss of a plasticizer.

A wettable Teflon® membrane revealed zero bubble pressure. This membrane consisted of a thin porous Teflon® film laminated onto a perforated backing. Post-test examination revealed that the low gas pressure applied at the startup had ruptured the porous Teflon® film covering several of the perforations.

The compatibility of an acrylic polymer/weak acid (NF-WA) microporous ion exchange membrane was concluded after several hours exposure to hot aqueous potassium hydroxide. The material was found to have such low strength, and general incompatibility with the electrolyte that a meaningful test could not be performed.

D. POROUS NAFION® MATRIX

A microporous Nafion® XR perfluorosulfonic acid resin sheet was developed several years ago for evaluation under the Power System Division commercial acid fuel cell program. The sheet was 10 mils (.25mm) thick, 80 percent porous and exhibited bubble pressures in excess of 60 psi (41.4 N/cm^2) when filled with aqueous potassium hydroxide.

Corrosion testing of the Nafion® revealed that samples of the material lost 13 percent of their weight after both 3000 and 5000 hours of exposure to 42% wgt. KOH at 250°F (121.1°C). However, scanning electron micrographs revealed heterogeneous attack, indicating an inhomogeneous composition.

Lewis Research Center identified a potential source for samples of improved porous Nafion®. A sheet of Teflon® screen reinforced microporous Nafion® perfluorosulfonic acid resin membrane, 2 ft (0.6m) by 1.5 ft (0.5m) by 20 mils (.5mm) thick was purchased.

A 3000-hour corrosion test at 250°F (121.1°C) of samples of the Teflon® reinforced membrane was conducted. Table XII presents corrosion test results.

Table XII. Nafion® Corrosion Data

Time-Hours	Weight Change - %
250	-5.4
500	-4.2
1000	-5.9
3000	-9.8

Although moderate weight loss was observed after 3000-hours of electrolyte exposure, there was no apparent loss in strength or deterioration in visual appearance. Test results indicated that the membrane was compatible with potassium hydroxide.

A research cell, RC-64 incorporating a Teflon® reinforced Nafion® membrane was constructed and evaluated. Unfortunately the performance level was unstable and there was a corresponding increase in cell internal resistance (iR). A test summary of RC-64 is presented in Appendix A.

There was no further work conducted under the program on Nafion® membranes.

E. BONDED POLYANTIMONIC ACID MATRICES

Lewis Research Center of NASA had suggested that polyantimonic acid (PPA) might be a candidate matrix material based upon its reported chemical stability in hot aqueous potassium hydroxide.

A sample of PPA was prepared in the laboratory from a procedure outlined in the literature. The material was similar in appearance to potassium titanate powder.

Two matrices were fabricated. One sample consisted of 5% wgt. Teflon® bonded PPA and the second sample employed asbestos as a reinforcing fiber. These samples were incorporated into laboratory Research Cells RC-81 and RC-82 and evaluated. A test summary is presented in Appendix A.

Cell performance for both cells was low and exhibited rapid fall-off with time. No further work with PPA matrices was conducted under the program.

A matrix structure with adequate bubble pressure and chemical stability will have to be fabricated before additional work with PPA matrices can be conducted.

F. POLYBENZIMIDAZOLE MATRICES

Polybenzimidazole (PBI) was identified as a candidate matrix material on the basis of low weight loss from corrosion tests in hot aqueous potassium hydroxide reported in NASA CR-159653. These tests, however, revealed considerable variation in test results and variation in physical makeup and color of samples. In addition endurance testing of research cells containing PBI matrices showed rapid voltage fall-off with time.

Since the initial evaluation, commercial grade PBI fibers have become available. Exploratory evaluation of matrices fabricated from commercial grade PBI fibers was conducted to re-evaluate the suitability of PBI for use in the fuel cell.

A free-standing, mat-type PBI matrix was fabricated in the laboratory and incorporated into Research Cell RC-78 for evaluation. A test summary of RC-78 is presented in Appendix A.

The inability to achieve acceptable performance characteristics was suspected to be due to poisoning of the catalyst by some decomposition product from the organic fiber. Test results reconfirm that PBI in the available forms is unsuitable for use in the alkaline fuel cell.

G. COMPOSITE MATRIX

The composite matrix consists of a potassium titanate layer deposited onto an advanced asbestos layer. A sketch of the alkaline fuel cell incorporating the composite matrix is shown on Figure 17. The asbestos layer provides structural integrity and the high bubble pressure.

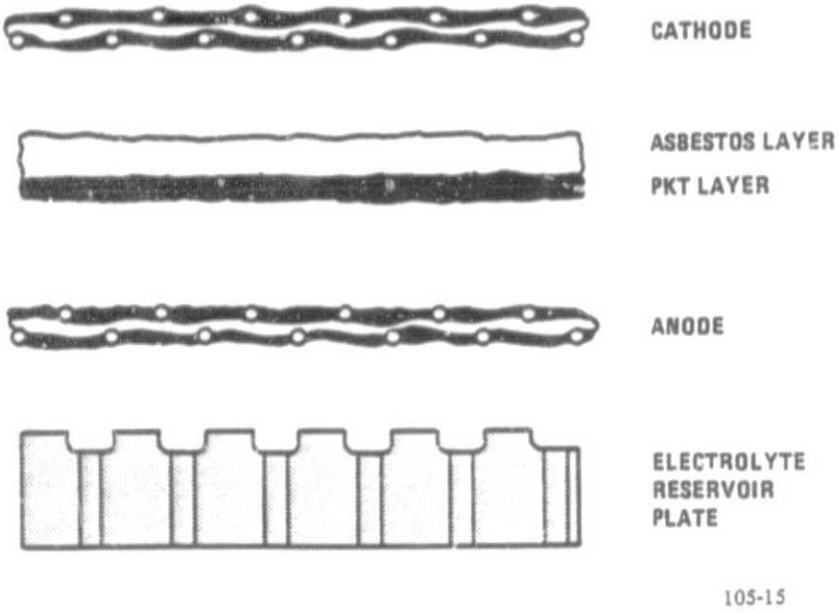


Figure 17. Composite Matrix Layer

Photomicrographs of long-term endurance cells which have exhibited a greater than expected performance fall-off with time have revealed an apparent electrochemical cleansing and material loss in the area adjacent to the anode with the standard asbestos matrix. The demonstrated stability of the potassium titanate layer in the composite matrix would not contribute any contaminates to the anode, thereby improving the long-term performance characteristics of the alkaline fuel cell.

Two composite matrices were fabricated and incorporated into Research Cells RC-69 and RC-71 and endurance tested. A summary of these research cell tests are presented in Appendix A.

Research cell RC-71 successfully completed a planned 5000-hour endurance test. Test results verify that the reduced silicon-content composite matrix is suitable for fuel cell use.

The development of a high bubble pressure, free standing, bonded potassium titanate matrix which is silicon free and has high temperature capability led to discontinuing work under the program on the composite matrix.

H. MATRIX EVALUATION PROCEDURES

Two of the most useful laboratory screening tests for evaluating candidate matrix materials are electrolyte compatibility and bubble pressure.

Electrolyte compatibility is a measure of a material's corrosion resistance with time to exposure at 250°F (121.1°C) in 42% wgt. aqueous potassium hydroxide.

Bubble pressure is a measure of the cross pressure at which gas breaks through the wetted matrix.

1. ELECTROLYTE COMPATIBILITY TEST PROCEDURE

The resistance of candidate matrix materials to electrolyte was determined by immersing the samples in 42 wgt. % potassium hydroxide at 250°C (121.1°C). the aqueous potassium hydroxide was contained in a Teflon® beaker in a sealed glass reaction kettle. The test apparatus is shown in Figure 18.

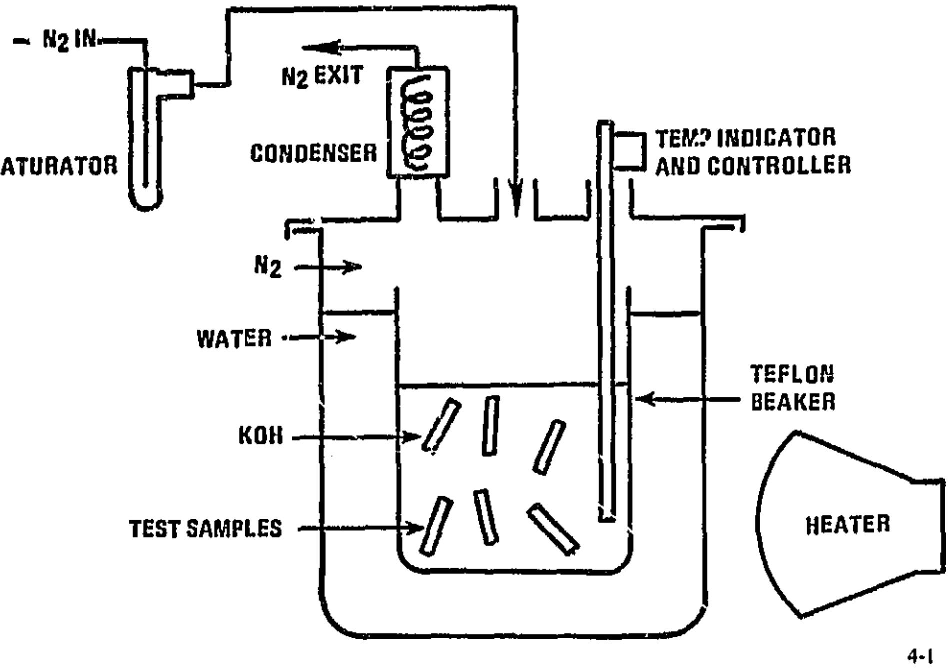


Figure 18. Material Corrosion Test Apparatus

Fixed quantities of each material are divided into four equal volumes and placed in 5-mil (0.1mm) folded Teflon® containment bags. Holes in the tops of each bag allow KOH free access to the sample. The four bags are then placed in a glass test vessel containing electrolyte sufficient for total immersion.

Water cooled condenser columns on each vessel maintain proper electrolyte concentration, while temperature is maintained by individual heat lamps with temperature controllers.

Time and temperature are both monitored by the Automatic Data and Recording (ADAR) system which is linked to a Cathode Ray Tube (CRT) display and printer. Temperatures are read at five-minute intervals, alerting laboratory technicians in the event temperature fall out of the ±3°F (±1.7°C) set point band.

Individual test samples are evaluated at 250, 500, 1000 and 3000-hour intervals. The evaluation consists of a visual inspection, gravimetric, electron microscopy and X-Ray diffraction analysis.

Corrosion test results form the basis of selecting candidate materials for further evaluation.

2. BUBBLE-PRESSURE MEASUREMENT TEST APPARATUS

The bubble-pressure measurement test apparatus is shown in Figure 19. The apparatus consists of two, 4 in. x 4 in. x 0.75 in (10.2 cm x 10.2 cm x 1.9 cm) plexiglass blocks. One block has a 3/8 in (0.95 cm) hole drilled through its center and is connected to a nitrogen supply by means of a threaded metal fitting. The other block has seventeen 1/8 in (0.3 cm) diameter holes drilled within a 1 in. (2.5 cm) diameter circle in the center of the block. The sample matrix supported on a nickel screen is centered between the two blocks. Rubber gaskets are utilized to seal off all but a 7/8 in. (2.2 cm) diameter portion of the matrix sample. The two blocks are clamped together with C-clamps and immersed in a water-filled container. The matrix sample is allowed to soak for 15-minutes to allow thorough wetting of the sample. The nitrogen gas is turned on and the pressure is gradually increased until either a breakthrough occurs, as evidenced by a steady stream of bubbles, or the pressure reaches 50 psi (34.5 N/cm^2), the pressure gage limit.

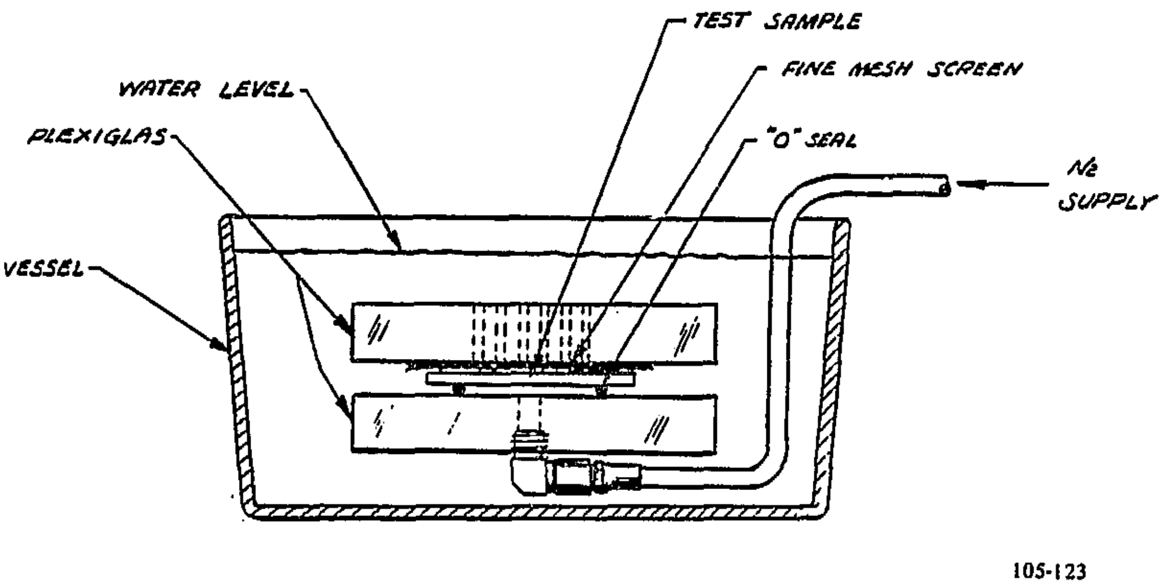


Figure 19. Bubble Pressure Measurement Test Apparatus

V. FUEL CELL ELECTRODE RESEARCH

The focus of the electrode research activity was to evaluate lightweight, low-cost electrode configurations with the potential to operate for 20,000-hours in a hydrogen-oxygen fuel cell operating at 60 psia (41.4 N/cm^2) reactant pressure and a cell temperature up to 250°F (121.1°C).

Endurance testing of laboratory research cells incorporating advanced anode and cathode configurations and matrix configurations developed under the program was conducted to demonstrate suitability for use in the alkaline fuel cell. In addition electrode fabrication and cell assembly techniques were evaluated to identify procedures which result in reproducible cell performance characteristics.

A platinum-on-carbon catalyst anode was evaluated under the program and has shown the potential for extended endurance.

A lightweight carbon ribbed substrate anode configuration which reduces standard production cell weight by nearly 50 percent has been successfully endurance tested.

Bonded potassium titanate matrices have shown stability in research cell endurance tests at cell temperatures up to 250°F (121.1°C). Two research cells incorporating potassium titanate matrices were tested for 1000-hours at 250°F (121.1°C).

Advanced matrix configurations consisted of bonded Ceria, bonded strontium titanate and lithium cobaltate were successfully incorporated into research cells and endurance tested.

A perforated nickel foil electrode substrate was designed, fabricated and endurance tested. This substrate has the potential for low cost, provides cell active area design flexibility and can be procured as a completed cell component not requiring in-house remanufacture.

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A hydrazine diluted electrolyte fill procedure was identified that contributed to reproducible platinum-on-carbon catalyst anode cell performance.

A nickel treatment of the carbon ribbed substrate anode was shown to promote the performance stability of cells incorporating this substrate during endurance testing.

A. LABORATORY RESEARCH CELL TEST FACILITY

A half-cell test fixture and a research cell test bench comprise the laboratory research cell test facility.

The half-cell test fixture consists of an Automatic Performance Evaluator, a Galvanostat-Potentiostat, associated glassware and a reactant flow and pressure control panel. In this fixture the performance of novel electrode configurations such as catalyst, substrate, and fabrication technique can be measured directly to a hydrogen reference electrode. The half-cell test also provides quick screening of experimental catalyst formulations and electrode configuration to identify the most promising for evaluation in laboratory research cell endurance tests.

In addition the laboratory half-cell test fixture is employed as a backup for the shop production test fixture, and is used to verify production data. The fixture is also employed to document electrode performance of cells that have completed long-term endurance tests.

The research cell test bench shown on Figure 20 can simultaneously endurance test up to four laboratory cells with future plans to expand this capability to eight cells. The endurance tests are conducted at ambient pressure at cell temperatures up to 250°F (121.1°C). The test bench electronic load box is of a special PSD design and has proven to be extremely reliable. As an example, in twelve years of continuous operation, the load box has been repaired once.

The test bench reactant systems and heater circuits have been designed to be as simple as possible to minimize components for improved operational reliability.

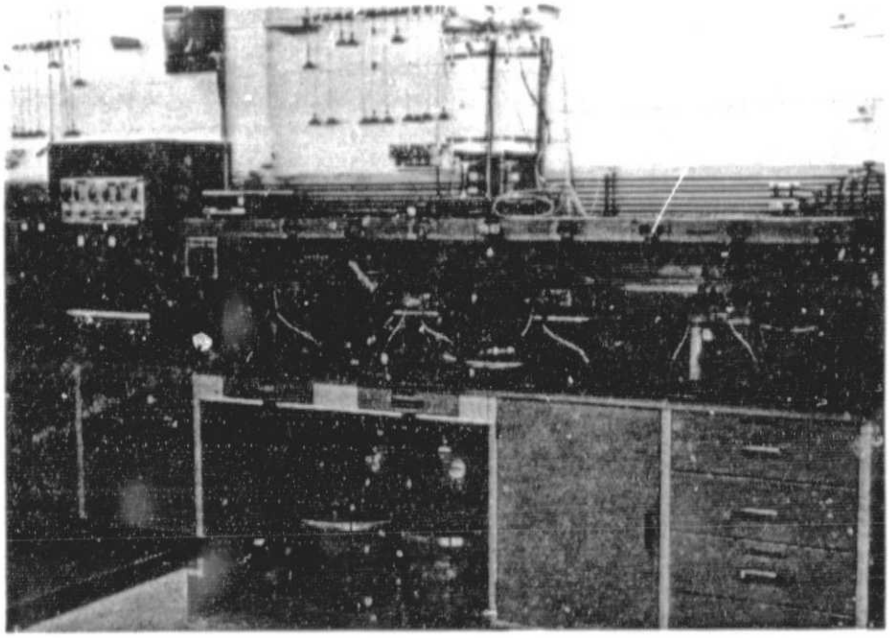

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Figure 20. Research Cell Test Facility

All reactants employed on the test bench are scrubbed to remove CO, CO_2 before introduction into the cells. Special reactant gas mixtures for periodic performance checkout tests are available on the test bench. In the event of the loss of utility line power the capability to automatically transfer to facility emergency power has been provided.

The laboratory test facility also has all the necessary equipment to manufacture electrodes in a size up to 0.508 square foot (471.9 cm^2). This capability permits quick evaluation of new full-size electrode configurations.

B. LABORATORY SUBSCALE RESEARCH CELL

This cell configuration was the fuel cell test vehicle for the exploratory evaluation of new cell components. Fuel cell technology advances evaluated in the research cell include:

- Bonded Potassium Titanate Matrices
- Asbestos Reinforced Potassium Titanate Matrices
- Bonded Particulate Matrices
- Composite Matrices
- Organic Membranes
- Lightweight Carbon Electrolyte Reservoir Plates
- Carbon Ribbed Substrate Anodes
- Gold-Plated Photo-Fabricated Nickel Foil Substrate Cathodes
- Platinum-on-Carbon Catalyst Anodes

A typical laboratory research cell test configuration is shown in Figure 21.

The cell has an active area of 0.028 ft^2 (25.8 cm^2) with planar dimensions of 2 inches x 2 inches (5.08 cm x 5.08 cm). Stainless steel end plates provides for manifolding of reactants and reactant flow fields are machined into the plates. A high pin coverage field is employed to minimize electrical resistance and current collector tabs are provided on each end plate for current collection. The end plates are gold-plated on all surfaces potentially exposed to potassium hydroxide to retard corrosion.

A non-unitized, Teflon® gasket cell edge frame is employed to provide a reactant seal and maintain cell package structural integrity. Normally the cells are constructed with 20-mil (0.5mm) thick matrices and 51-mil (1.3mm) thick porous nickel electrolyte plates in contact with the anode.

The cells operate with dynamic water removal, that is, cell product water is removed by flowing excess hydrogen.

A summary of the research cells tested during the program is presented in Section V.

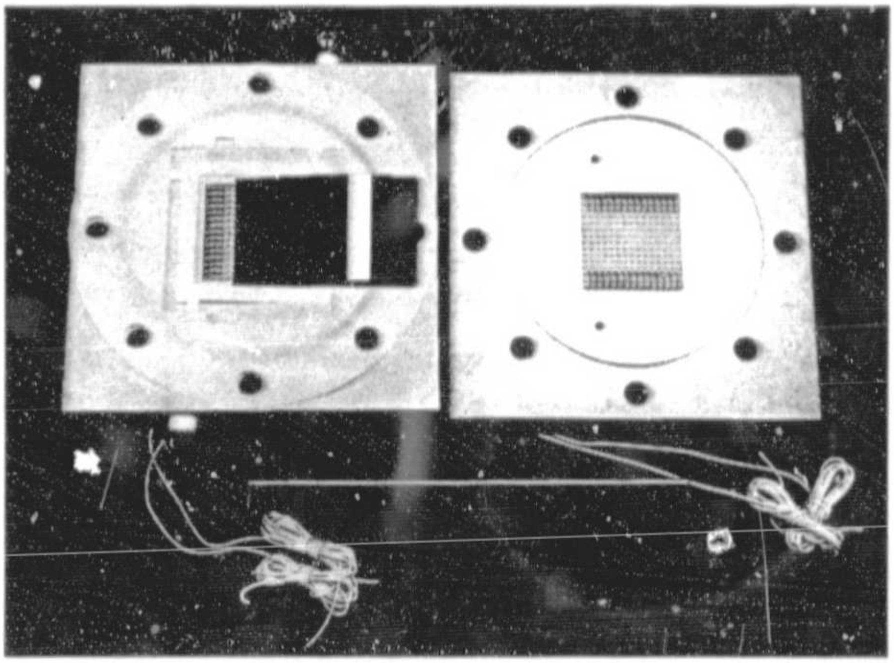

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Figure 21. Typical Research Cell Hardware Test Setup

C. RESEARCH CELL TEST SUMMARY

A total of 64 research cells were fabricated and endurance tested. These tests focused upon the evaluation of cells incorporating:

- Platinum-on-Carbon Catalyst Anodes
- Carbon-Ribbed Substrate Anodes
- Bonded Potassium Titanate Matrices
- Advanced Matrix Configurations
- Perforated Nickel Foil Electrode Substrate
- Improved Electrode Fabrication and Cell Assembly Procedures

Table XIII summarizes the test results of the 56 research cells that have been tested. Appendix A presents a discussion of individual research cell endurance test results.

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-40	90 Au-10 Pt (Au-Plated Ni Screen)	2,7-mil ASB with 5% Binder	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	1116	Test Completed
RC-41	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt Pd (Ribbed Carbon Substrate)	N/A	292	Test Completed
RC-42	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt Pd (Au-Plated Ni Screen Selectively Wet-Proofed)	51-mil Ni	2136	Test Completed
RC-43	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Ribbed Carbon Substrate)	N/A	25	Test Stopped Low Performance
RC-43A	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Ribbed Carbon Substrate)	N/A	338	Test Stopped. Low Performance
RC-44	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Graphite Impregnated Ribbed Carbon Substrate)	N/A	295	Test Stopped. Low Performance
RC-45	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Ribbed Carbon Substrate Selectively Wet-Proofed)	N/A	200	Test Stopped. Internal Cell Short
RC-46	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Nickel Treated Ribbed Carbon Substrate)	N/A	648	Test Stopped. Low Performance

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-47	90 Au-10 Pt (Au-Plated Ni-Screen)	20-mil ASB (Reconstituted)	Pt/C (Nickel Treated, Ribbed Carbon Substrate, Selectively Wet-Proofed)	N/A	278	Test Stopped. Low Performance
RC-48	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Au-Plated Photo-fabricated Ni foil)	100-mil Ni	300	Test Stopped. Low Performance
RC-48A	90 Au-10 Pt (Au-Plated Ni Screen)	20 mil ASB (Reconstituted)	Pt/C Au-Plated Photo-fabricated Ni foil)	100 mil Ni	37	Test Stopped. Low Performance
RC-49	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Ni-treated Ribbed Carbon Substrate with Holes)	N/A	1250	Test Complete
RC-50	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt Pd (Ni-treated Ribbed Carbon Substrate with Holes)	N/A	1285	Test Complete
RC-51	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil ASB (Reconstituted)	Pt/C (Carbon Paper Substrate	Ni-Treated Ribbed Carbon Substrate	166	Test Stopped. Low Performance
RC-52	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil 95% PKT 5% TFE	Pt Pd (Au-Plated Ni Screen)	50-mil Ni	2225	Test Completed

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-53	90 Au-10 Pt (Ni/CO-Treated Feltmetal® Substrate)	20-mil ASB (Reconstituted)	P&PD (Au-Plated Ni Screen)	50-mil Ni	174	Test Stopped. Low Performance
RC-54	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil 96% PKT 4% Butyl Rubber	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	5025	Test Complete
RC-55	90 Au-10 Pt (Au-Plated Ribbed Nickel Feltmetal®)	20-mil ASB (Reconstituted)	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	1007	Test Complete
RC-56	Uncatalyzed (Au-Plated Ribbed Nickel Feltmetal)	20-mil ASB (Reconstituted)	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	2	Test Stopped. Low Performance
RC-57	90 Au-10 Pt (Au-Plated Ribbed Nickel Feltmetal)	20-mil ASB (Reconstituted)	Pt Pd (Ni-Treated Ribbed Carbon Substrate with Holes)	N/A	500	Test Stopped. Cell Hardware Required
RC-58	90 Au-10 Pt (Au-Plated Perforated Ni Foil)	20-mil ASB (Reconstituted)	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	938	Test Complete
RC-59	90 Au-10 Pt (Au-Plated Perforated Ni Foil)	20-mil ASB (Reconstituted)	Pt/C (Au-Plated Perforated Ni Foil)	51-mil Ni	160	Test Stopped. Stand Relocation
RC-60	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil 96% PKT 4% Butyl Rubber	Pt Pd (Au-Plated Ni Screen)	51-mil Ni	1135	Test Complete 250°F(121.1°C)

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-61	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil 95% PKT 5% TFE	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	1135	Test Complete 250°F(121.1°C)
RC-62	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil 95% PKT 5% TFE	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	5110	Test Complete
RC-63	90 Au-10 Pt (Au-Plated Perforated Ni Foil)	20-mil Advanced Asbestos	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	1171	Test Stopped (low performance)
RC-64	90 Au-10 Pt (Au-Plated Ni Screen	20-mil Micro porous Nafion®	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	241	Test Stopped (low performance)
RC-65	90 Au-10 Pt (Au-Plated Perforated Ni-Foil)	20-mil Advanced Asbestos	Pt Pd (Au-Plated Ni Screen)	51-mil Porous	1020	Test Complete
RC-66	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt Pd (Dual Porosity Carbon Ribbed Substrate)	N/A	1010	Test Complete
RC-67	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Ni-treated Carbon Ribbed Substrate)	N/A	1300	Test Complete
RC-68	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Thin Web Dual Porosity Ni-Treated Carbon Ribbed Substrate)	N/A	806	Test Stopped. (Low Performance)

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-69	90 Au-10 Pt (Au-Plated Ni Screen)	Composite (Asbestos/ Potassium Titanate)	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	500	Test Stopped (low performance)
RC-70	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Dual Porosity, Ni Treated Carbon Ribbed Substrate)	N/A	665	Test Stopped (low voltage)
RC-71	90 Au-10 Pt (Au-Plated Ni Screen)	Composite (10-mils Asbestos, 10-mils Potassium Titanate)	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	5110	Test Completed
RC-72	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Ni-Treated Carbon Ribbed Substrate)	N/A	237	Test Stopped (low voltage)
RC-73	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Ni-Treated Carbon Substrate)	51-mil Porous Ni	1300	Test Stopped (low voltage)
RC-74	90 Au-10 Pt (Au-Plated Perforated Ni Foil)	20-mil Advanced Asbestos	Pt Pd (Au-Plated Ni Screen)	51-mil Porous Ni	1055	Test Complete
RC-75	90 Au-10 Pt (Au-Plated Ni Screen)	20-mil Advanced Asbestos	Pt/C (Au-Plated Ni Screen)	51-mil Porous Ni	345	Test Complete

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-76	90 Au-10 Pt (Au-Plated Ni Screen)	7-mils Butyl Rubber Bonded Ce O_2	Pt Pd (Au-Plated Ni Screen)	51-mils Porous Ni	496	Test Stopped (low voltage)
RC-77	90 Au-10 Pt (Au-Plated Ni Screen)	6-mils Butyl Rubber/ Strontium Titanate	Pt Pd (Au-Plated Ni Screen)	51-mils Porous Ni	5200	Test Completed
RC-78	90 Au-10 Pt (Au-Plated Ni Screen)	Lab Made PBI Mat	Pt Pd (Au-Plated Ni Screen)	51-mils Porous Ni	1205	Test Stopped (low voltage)
RC-79	90 Au-10 Pt (Au-Plated) Ni Screen	20-mil Advanced Asbestos	Pt Pd (Ni-Treated) Dual Porosity Carbon Ribbed Substrate	N/A	1300	Test Completed
RC-80	90 Au-10 Pt (Au-Plated) Ni Screen	20-mil Advanced Asbestos	Pt Pd (Ni-Treated) Dual Porosity Carbon Ribbed Substrate	N/A	1200	Test Completed
RC-81	90 Au-10 Pt (Au-Plated) Ni Screen	Polyantimonic Acid 5% Teflon®	Pt Pd (Au-Plated Ni Screen)	51-mils Porous Ni	1	Test Stopped. (reactant cross-leakage)
RC-82	90 Au-10 Pt (Au-Plated) Ni Screen	80% Polyantimonic Acid/20% Asbestos	Pt Pd (Au-Plated Ni Screen)	51-mils Porous Ni	6	Test Stopped (low voltage)
RC-83	90 Au-10 Pt (Au-Plated) Ni Screen	92% Potassium Titanate/ 8% Butyl Rubber	Pt/C (Au-Plated Ni Screen)	51-mils Porous Ni	184	Test Stopped (low voltage)

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-84	90 Au-10 Pt (Au-Plated) Ni Screen	92% Potassium Titanate/ 8% Butyl Rubber	Pt/C (Au-Plated Ni Screen	51-mils Porous Ni	50	Test Completed
RC-85	90 Au-10 Pt (Au-plated) Ni Screen	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	1035	Test Completed
RC-86	90 Au-10 Pt (Au-plated) Ni Screen)	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	1155	Test Completed
RC-87	90 Au-10 Pt (Au-plated) Ni Screen	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	500	Test Completed
RC-88	90 Au-10 Pt (Au-plated) Ni Screen	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	NA	505	Test Completed
RC-89	90 Au-10 Pt (Au-plated) Ni Screen	20-mil Advanced Asbestos	Pt/Pd (Au-plated Ni Screen with Carbon Layer	51-Mil Ni	1159	Test Continues
RC-90	90 Au-10 Pt (Au-plated) Ni Screen	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	802	Test Continues
RC-91	90 Au-10 Pt (Au-Plated) Ni Screen	20-mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	292	Test Stopped (low voltage)

Table XIII. Research Cell Test Summary

Cell No.	Cathode	Matrix	Anode	ERP	Test Hours	Comments
RC-92	90 Au-10 Pt (Au-Plated) Ni Screen	96% $LiCoO_3$ 4% Butyl Rubber	Pt Pd (Au-Plated Ni Screen)	51-Mil Ni	1	Test Stopped (low voltage)
RC-93	90 Au-10 Pt (Au-Plated) Ni Screen	20-Mil Advanced Asbestos	Pt/C (Ni-Treated Dual Porosity Carbon Substrate)	N/A	141	Test Continues

1. Platinum-On-Carbon Catalyst Anodes

The supported catalyst has an advantage over the unsupported catalyst configurations in two important aspects. In dispersing platinum uniformly over the surface of an inert support, smaller crystallites of the noble metal are obtained and therefore, a significant higher specific surface area, meter square per gram, is possible compared to an unsupported configuration. The second advantage is that the support material, being a structural member of the catalyst layer maintains the integrity of the electrode even if the catalyst particles sinters or recrystallize.

Endurance testing of platinum-on-carbon catalyst anode cells in a previous program (Reference 8) has shown that the 0.5 mg/cm² platinum supported-on-carbon anode, 10% platinum to carbon has potential for extended endurance. Based upon research cell test results, this anode configuration was incorporated into the long-term six-cell stack endurance test rig 39578 which has completed 18,054 hours of cyclical load profile operation.

Typical performance of platinum-on-carbon catalyst anode cells is presented in Figure 22.

The focus of the work under this program was to develop a platinum-on-carbon catalyst layer deposited onto a carbon ribbed substrate anode. This configuration has the advantages of being lightweight and low-cost and offers the potential for long-life. Twenty-five research cells were fabricated and endurance tested. Test results from these cells have been encouraging and are showing promise that this anode configuration is a viable candidate for use in the alkaline fuel cell.

2. Carbon-Ribbed Substrate Anodes

The porous ribbed anode substrate, provides a single component which provides not only reactant gas flow fields, but serves as a substrate for the anode catalyst and an electrolyte reservoir. Since the substrate is carbon, it is both light and inexpensive particularly when compared to the gold-plated separator, nickel electrolyte reservoir plate and gold-plated nickel screens used in the standard production cell.

The substrate consists of a porous carbon plate which has a parallel rib pattern machined into one surface. The free area between the ribs serve as the hydrogen flow field with a stiffening web between ribs. The electrode is formed by depositing the catalyst layer onto the flat side of the substrate.

A photograph of the carbon-ribbed substrate is shown on Figure 23.

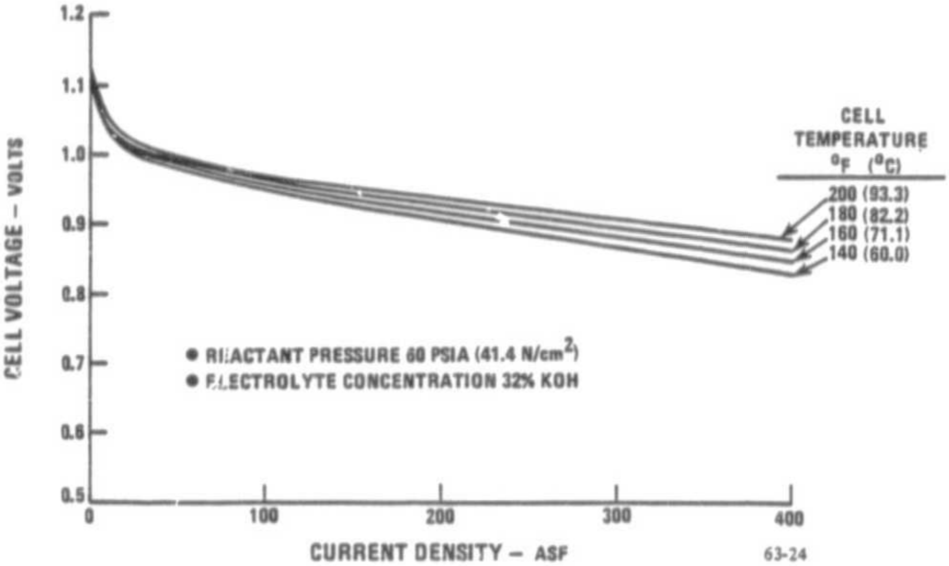

Figure 22. Typical Platinum-on-Carbon Catalyst Anode Cell Performance

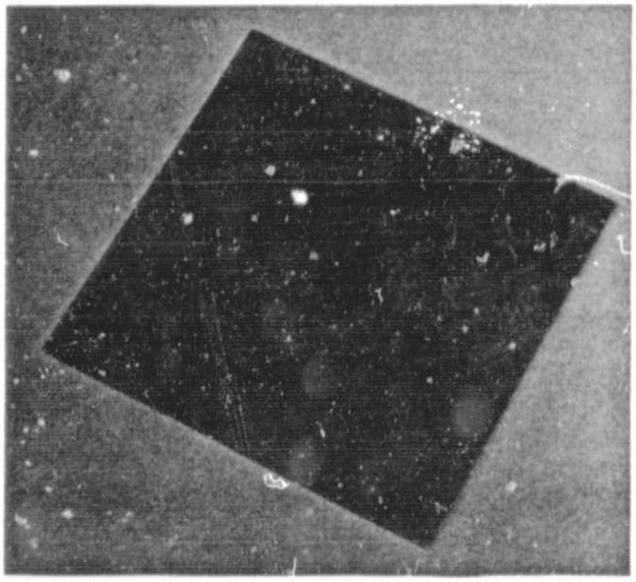

Figure 23.
Typical Carbon Ribbed Substrate

(WCN-11030)

Two varieties of dual porosity substrates were evaluated under the program. In one configuration the ribs were about twice the density of the web and in the second configuration the ribs and web have similar densities. Both types of substrated are treated with nickel to render them wettable prior to applying the catalyst layers.

Twenty-four research cells incorporating carbon ribbed substrate anodes were fabricated and endurance tested. The single porosity substrates are showing promise as a potential lightweight, low-cost substitute for use at the anode in present alkaline fuel cell powerplants. Additional work is in progress to identify a substrate with a smaller mean pore size which will retain electrolyte during launch. Tolerance to electrolyte volume variation also needs to be demonstrated.

3. Bonded Potassium Titanate Matrices

Laboratory research cell endurance testing of Butyl rubber and Teflon® bonded potassium titanate matrices has demonstrated their suitability for use in the alkaline fuel cell.

Significant long-term research cell test results are summarized in Table XIV.

Table XIV. Bonded Potassium Titanate Matrix Research Cell Test Summary

Research Cell	Matrix Compound	Cell Temp °F	(°C)	Test Hours
52	5% TFE/95% PKT	200	(93.3)	2225
54	4% Butyl/96% PKT	200	(93.3)	5025
60	4% Butyl/96% PKT	250	(121.1)	1135
61	5% TFE/95% PKT	250	(121.1)	1135
62	5% TFE/95% PKT	200	(93.3)	5110

All of the endurance tests presented in Table XIV were stopped following completion of all test objectives.

In addition endurance testing has demonstrated the capability of the matrix configuration to operate at high temperature, up to 250°F (121.1°C).

4. Advanced Matrix Configurations

Six advanced matrix configurations identified under the program and described in Section IV-A were incorporated into laboratory research cells and endurance tested. The objective of these tests was to evaluate the effect of the matrix upon fuel cell performance stability. The six configurations were:

Matrix Configuration	Research Cell No.
• Butyl Bonded Ceria	RC-76
• Butyl Bonded Strontium Titanate	RC-77
• Butyl Bonded Lithium Cobaltate	RC-92
• Porous Nafion®	RC-64
• Polybenzimidazole	RC-78

The research cells incorporated standard production anodes, cathodes and porous nickel electrolyte reservoir plates. Therefore any deviation in cell performance would be attributable to the matrix. The cells were endurance tested at 200 Amps/Ft^2 (215.3 mA/cm^2), 200°F (93.3°C) and ambient pressure.

A summary of test results is presented in the following discussion.

Butyl Bonded Ceria

The endurance test of the bonded Ceria cell (RC-76) was stopped after 496-hours due to a greater than expected voltage reduction. Prior to the commencement of the test, visual inspection identified a very dense, with some cracks, marginal matrix structure. Additional matrix fabrication trials will be required to arrive at a structure suitable for research cell testing.

Butyl Bonded Strontium Titanate

A research cell (RC-77) incorporating a bonded strontium titanate matrix successfully completed a 5000-hour endurance test.

A review of cell performance data indicated that the majority of the voltage loss was due to an increase in anode polarization. A possible explanation for increased anode polarization is that some unbound particulate may have penetrated the catalyst layer promoting partial flooding of the electrode.

Strontium titanate, because of the chemical similarity to potassium titanate and research cell test results, has potential to be a candidate matrix material. Additional matrix fabrication required to improve the structure.

Butyl Bonded Lithium Cobaltate

The initial performance of the research cell (RC-92) incorporating a bonded $LiCoO_3$ matrix was low due to higher than expected internal resistance. After only an hour of testing gas cross leakage occurred and the endurance test was stopped.

Based upon test results from this exploratory test, $LiCoO_3$ is unsuitable for use as a matrix material in the alkaline fuel cell.

Porous Nafion®

The performance of the research cell (RC-64) containing porous Nafion® matrix was very low due to high internal resistance. During testing, cell voltage fell off with time with a corresponding increase in cell iR.

Test results and teardown inspection of the research cell has shown that porous Nafion® is unsuitable as a matrix material.

Polybenzimidazole

An exploratory test of commercial grade polybenzimidazole (PBI) as a matrix material was conducted. Research cell endurance test results duplicated previous test experience (Reference 6). A research cell (RC-78) incorporating a PBI matrix exhibited rapid performance fall-off with time. The material is considered unsuitable for use as a matrix in the alkaline fuel cell.

5. Alternate Asbestos Matrix Configurations

As an approach to improving the long-term stability of the high strength asbestos matrix, three alternate configurations were evaluated. Each of the configurations, identified below, were incorporated into laboratory research cells and endurance tested.

- Advanced Asbestos
- Butyl Rubber Bonded Asbestos
- Composite Matrix

The research cells incorporated standard production anodes, cathodes and electrolyte reservoir plates. A summary of test results are presented in the following discussion.

Advanced Asbestos

The advanced asbestos matrix configuration was developed by Power Systems Division under the Orbiter Fuel Cell contract with Rockwell International. Carbonate analysis of endurance test cells reported in NASA CR-165417 revealed that the advanced asbestos matrix cells had less than half the carbonate level of standard asbestos matrix cells.

Advanced asbestos matrix has been incorporated into the cells being fabricated for the Orbiter Fuel Cell powerplant. In addition, the advanced asbestos matrix is the baseline configuration for all cell tests conducted under the Lewis program.

Butyl Rubber Bonded Asbestos

Lewis Research Center of NASA supplied Power Systems Division with a sheet of fuel cell grade asbestos containing approximately 5% butyl rubber binder. The matrix was incorporated into research cell RC-40 and successfully completed a planned 1000-hour endurance.

The stable performance characteristics observed during the endurance confirmed corrosion test results that the butyl rubber was suitable for use as a matrix binder in the alkaline fuel cell.

Composite Matrix

The composite matrix consists of a potassium titanate layer deposited onto an advanced asbestos layer. Two laboratory research cells, RC-69 and RC-71 incorporating composite matrices and standard production anodes, cathodes and electrolyte reservoir plates were endurance tested.

The endurance tests were characterized by lower than expected initial performance and increased anode polarization with time. Electrode flooding or contamination of the electrode with time are suspected of contributing to the unacceptable performance characteristics. Additional work would be required to identify a suitable composite matrix structure.

6. ALTERNATE ELECTRODE SUBSTRATES

A porous ribbed nickel and perforated nickel foil structure were evaluated as candidate electrode substrates.

Porous Ribbed Nickel

A cell incorporating an electrolyte reservoir (ER) adjacent to the anode and cathode has the potential for improved operational reliability. With this cell design, any electrolyte expelled during reactant cross pressure incidents would be absorbed by the ER. Upon return to normal operating conditions, the electrolyte would be transferred back into the cell.

A photograph of the porous ribbed nickel substrate and electrolyte reservoir is shown on Figure 24.

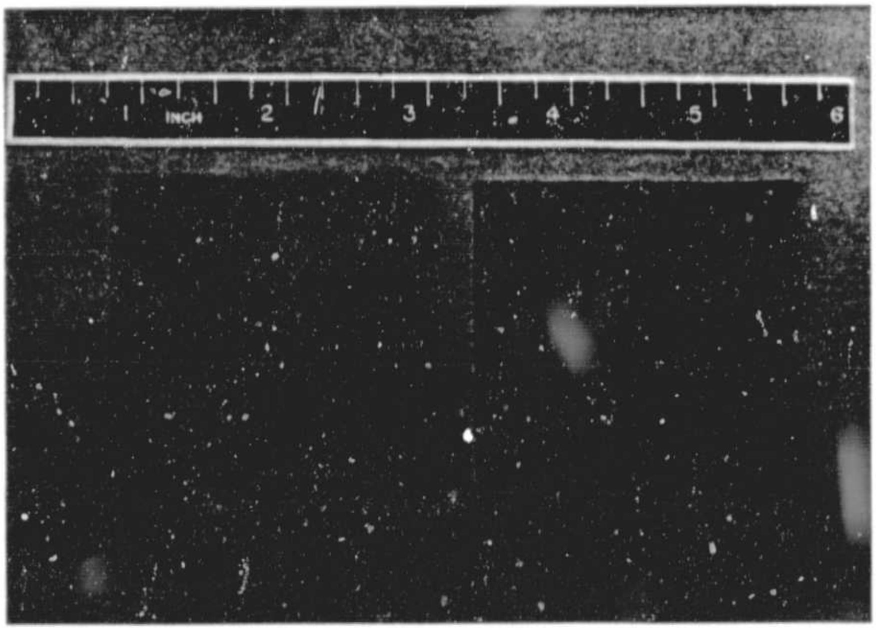

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Figure 24. Porous Ribbed Nickel Substrate

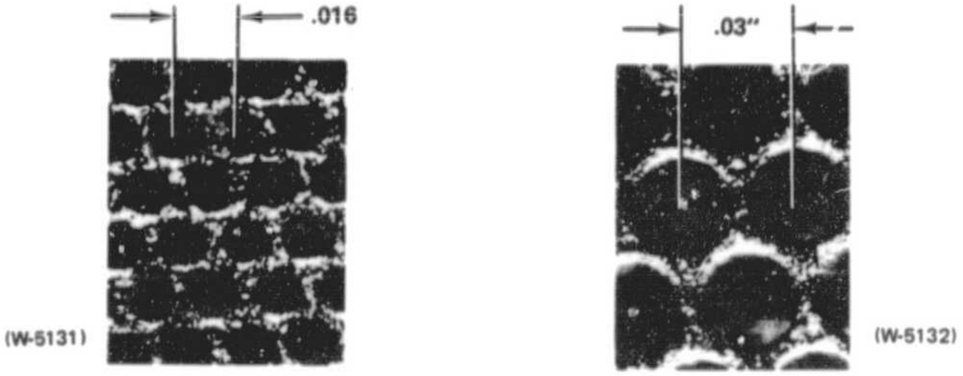
C-2

The electrode is formed by depositing a catalyst layer onto the flat surface. The ribbed surface forms the reactant flow field.

Test results from four laboratory research cells indicate that the ribbed nickel substrate cathode is stable at cathode potentials even with stored electrolyte, and does not contribute any additional diffusional loss, when compared to the standard nickel screen substrate.

Perforated Nickel Foil

The perforated nickel foil substrate has the potential to contribute to a substrate reduction in cost, provide design flexibility and possibly reduce cell weight. Figure 25 shows typical configurations of the photo-fabricated nickel foil substrates.



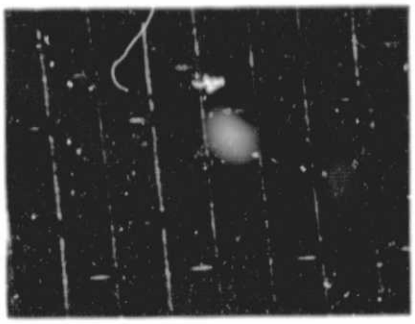
11 MIL DIAMETER HOLE

25 MIL DIAMETER HOLE

Figure 25. Perforated Photofabricated Nickel Foil Substrate

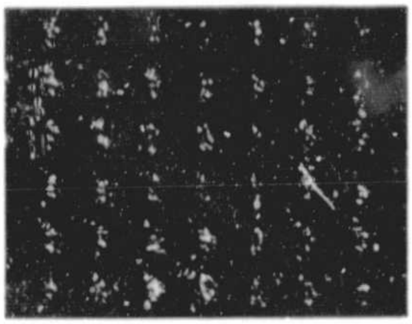
Laboratory research cell endurance testing of cells incorporating gold-plated nickel photo-fabricated foil substrates has shown that the foils are an acceptable replacement for the expensive gold-plated, fine-wire nickel screen presently employed in production cells.

Figure 26 shows a comparison of the gold-plated perforated nickel foil and standard gold-plated nickel screen substrate.



**STANDARD GOLD-PLATED
Ni SCREEN**

(W-5106)



**GOLD-PLATED NICKEL
PHOTO-FABRICATED FOIL**

(W-5107)

Figure 26. Electrode Substrate Comparison

In the evaluation of perforated nickel foil substrates, a total of four research cells (RC-58, RC-63, RC-65 and RC-74) were constructed and endurance tested. These cells completed over 4300-hours of testing.

7. SELECTIVELY WET-PROOFED ANODE CONFIGURATION

Selective wet-proofing of the hydrogen electrode consists of covering the gas-side with a thin film of porous Teflon® except where the nubbins of the electrolyte reservoir plate make contact. The selectively wet-proofed Teflon®-pattern screen-printed on the anode is shown on Figure 27.

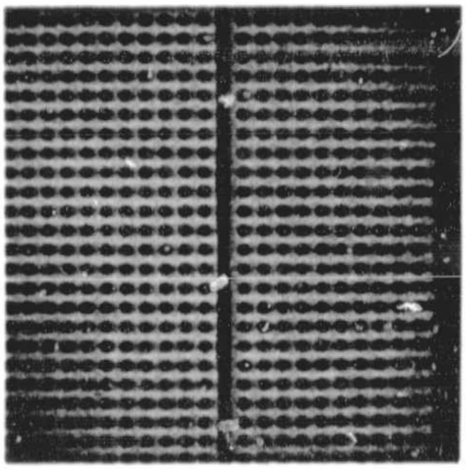


Figure 27. Selectively Wet-Proofed Anode Configuration

The function of the selective wet-proofing layer is to retard the formation of an electrolyte film on the reactant side of the electrode. This film would create a gas-diffusion barrier which contributes to lower anode performance.

Research cell endurance testing (RC-42, RC-45, and RC-47) has indicated that the selectively wet-proofed anode configuration improves cell tolerance to electrolyte volume variations resulting from changes in operating conditions.

The advent of the very hydrophobic platinum-on-carbon catalyst anode led to suspending work on wet-proofed anodes. The platinum-on-carbon anode with its good electrolyte retention characteristics has demonstrated extended endurance capability.

APPENDIX A

RESEARCH CELL TESTING

APPENDIX A

RESEARCH CELL TESTING

Exploratory evaluation of new anode and cathode configurations, and advanced matrix configurations was conducted with sub-scale laboratory research cells. A complete description of the laboratory research cell is presented in Section V. The cell active area is 0.028 ft^2 (26.0 cm^2) with planar dimensions of 2 inches x 2 inches (5.1 cm x 5.1 cm).

The majority of the laboratory research cell endurance tests was conducted at a cell temperature of 200°F (93.3°C), a reactant pressure of 14.7 psia (10.1 N/cm^2) and a current density of 200 amperes per ft^2 (215.3 mA/cm^2).

A total of 64 research cells were constructed and endurance tested during the program completing 51,777 hours of testing. A summary of the individual research cells tested is shown in Table XIII, Section V.

Test results of the individual research cells is presented in the following discussion.

RC-40 (Butyl Rubber bonded Asbestos Matrix)

Lewis Research Center of NASA provided Power Systems Division with a sheet of 7 mil thick fuel cell grade asbestos containing approximately 5% Butyl Rubber binder. A double layer of the mat, approximately 14 mils (.36mm) was built into a research cell and endurance tested.

The performance history of RC-40 is presented on Figure 28. RC-40 completed 1116-hours of testing with very little voltage fall-off during the test. The initial cell voltage was 0.922 V at 200 ASF (215.3mA/cm^3) and after 1116-hours cell voltage was 0.916 V. The endurance test was stopped upon completion of the planned 1000-hour test.

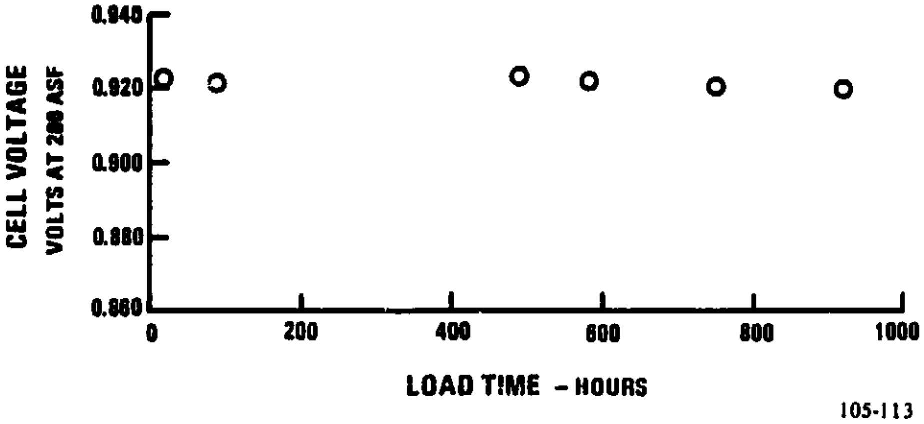


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Figure 28. Research Cell (RC-40) Endurance Test (Butyl Rubber Bonded Asbestos Matrix)

Test results indicate that butyl rubber is suitable for use as a matrix binder in alkaline fuel cells.

The characteristics (thickness, density and bubble pressure) were determined prior to the commencement of the endurance test. Table XV lists the characteristics of the butyl rubber bonded asbestos sheet.

Table XV. Butyl Rubber Bonded Asbestos Matrix Characteristics

●	Thickness	0.007 ± .003 in. (0.18 ± .08mm)
●	Density	0.767 gms/cc
●	Bubble Pressure	
	Single Ply - 7 mils	20 psid (13.8 N/cm^2)
	Double Ply - 14 mils	25 psid (17.2 N/cm^2)

For comparison, the bubble pressure of a Power Systems Division fabricated 20-mil (.5mm) asbestos matrix has demonstrated bubble pressure in excess of 50 psid (34.5 N/cm^2).

RC-41 (Carbon Ribbed Substrate Anode)

The individual cells in the Orbiter Fuel cell powerplant are unitized, that is, the electrodes matrix and electrolyte reservoir plate are bonded together at the edges by an epoxy-fiberglass cell edge frame. This frame forms a very effective electrical insulating seal and reactant pressure seal. Sealing difficulties experienced on earlier research cell tests, lead to the evaluation of the Orbiter edge seal approach in laboratory cells.

In the construction of RC-41, only the reconstituted asbestos matrix (RAM) and the gold-platinum catalyst cathode were unitized. The anode was a carbon ribbed substrate with a platinum-palladium catalyst layer.

The performance history of RC-41 is presented on Figure 29.

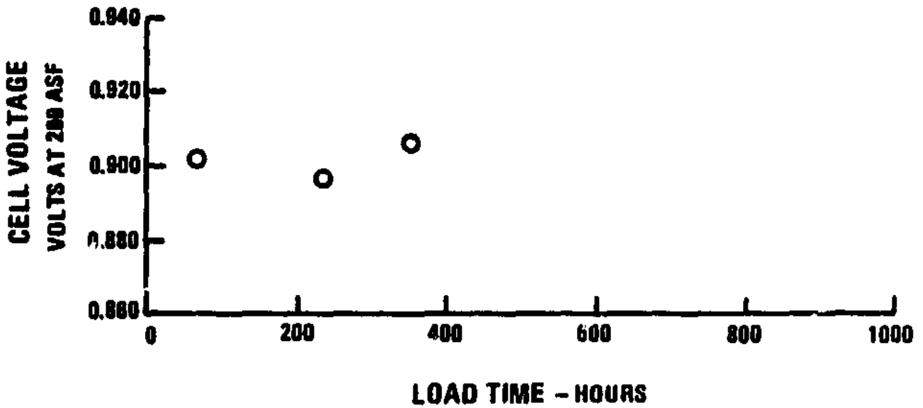

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Figure 29. Research (RC-41) Endurance Test (Carbon Ribbed Substrate Anode)

The cell was initially flush-filled with 23% wgt. KOH at room temperature. The initial performance of 0.905V at 200 ASF (215.3 mA/cm²) was reasonably good, but there was a greater than expected voltage fall-off initially. On the assumption that RC-41 had not been adequately wetted with electrolyte, the cell was removed from the test facility and refilled with 23% wgt. KOH containing a small amount of hydrazine. This electrolyte fill technique has proven successful in filling past supported-platinum catalyst anode cells.

On resumption of the endurance, the cell performance was restored to 0.905V at 200 ASF (215.3 mA/cm²) and a significant reduction in the rate of voltage fall-off. At 242 load hours, cell performance was 0.885V. At this point a second electrolyte refill was performed.

Upon return to endurance test condition, cell performance was again restored, 0.905V at 200 ASF (215.3 mA/cm²) which remained constant until the endurance test was stopped at 292-load hours due to a facility vacation shutdown.

Sealing difficulties with RC-41 were minimized, resulting in the conclusion that the partial unitization was successful. Test results indicate that the carbon ribbed substrates are reluctant to wet with electrolyte spontaneously. Subsequent research cell tests, evaluated substrate fabrication techniques which promoted electrolyte wetting of the structure.

RC-42 (Selectively Wetproofed Anode)

Under an earlier NASA-Lewis sponsored fuel cell technology advancement program (Reference 4) research cell test results suggested that performance reduction with time was caused by progressive electrolyte flooding of the platinum-palladium catalyst layer which might be alleviated by selectively wet-proofing the reactant side of the anode catalyst.

Selective wet-proofing of the hydrogen electrode consists of covering the gas side with a thin film of porous Teflon® except where the nubbins of the electrolyte reservoir plate make contact. Anodes incorporating photo-fabricated nickel foil substrates probably benefit from selective wet-proofing as this electrode configuration operates with a separate electrolyte reservoir plate.

The function of the selective wet-proofing layer is to retard the formation of an electrolyte film on the reactant side of the electrode. This film would create a gas-diffusion barrier which contributes to lower anode performance.

In order to define a wet-proofing procedure, even though photo-fabricated nickel foil had not yet been received from the vendor, a selectively wetproofed platinum-palladium catalyst anode was fabricated. This electrode was built into RC-42 and endurance tested in the laboratory test facility.

The performance history of RC-42 is presented on Figure 30.

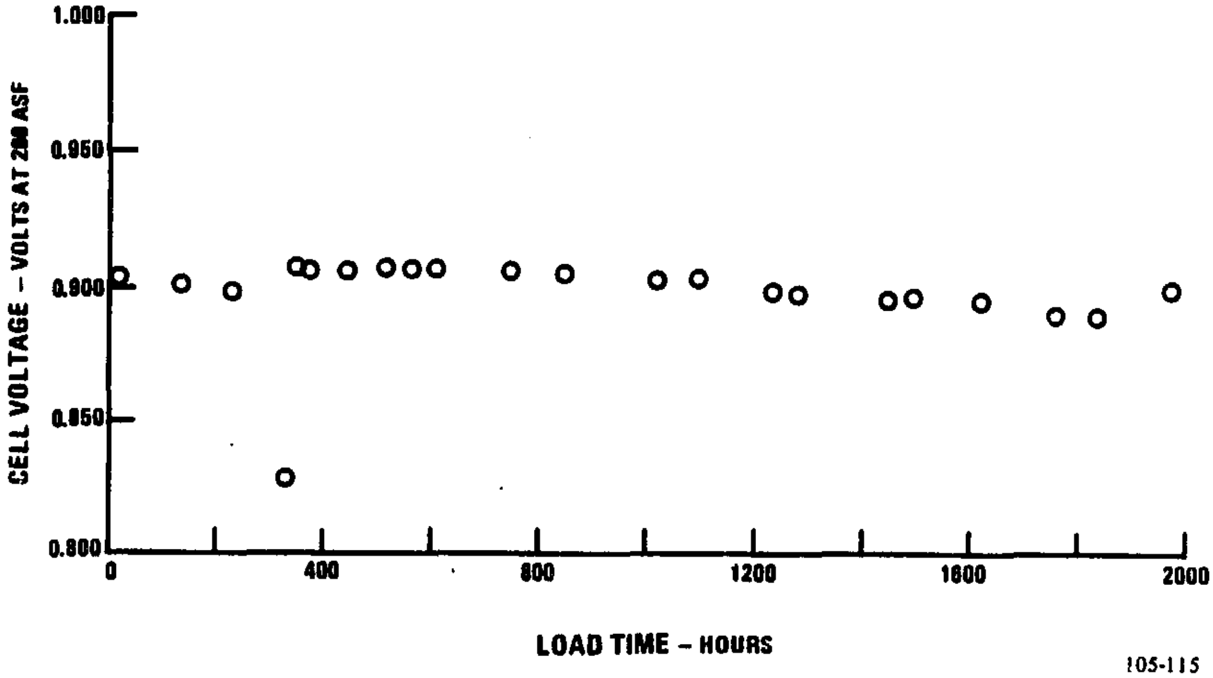

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Figure 30. Research Cell (RC-42) Endurance Test
(Selectively Wet-Proofed PtPd Catalyst Anode)

RC-42 was operated on unhumidified reactants, at a current density of 200 ASF (215.3 mA/cm²) a cell temperature of 200°F (93.3°C) and ambient pressure for the first 1840-hours of testing. The initial cell performance was 0.902V and after 1840 hours of testing cell voltage was 0.888 volts.

Retarding the formation of an electrolyte film on the electrode reactant surface would improve the performance stability of the cell to load and electrolyte volume changes. To evaluate the stability of RC-42 to these conditions the cell was subjected to the daily "stress test" cycle shown on Figure 31.

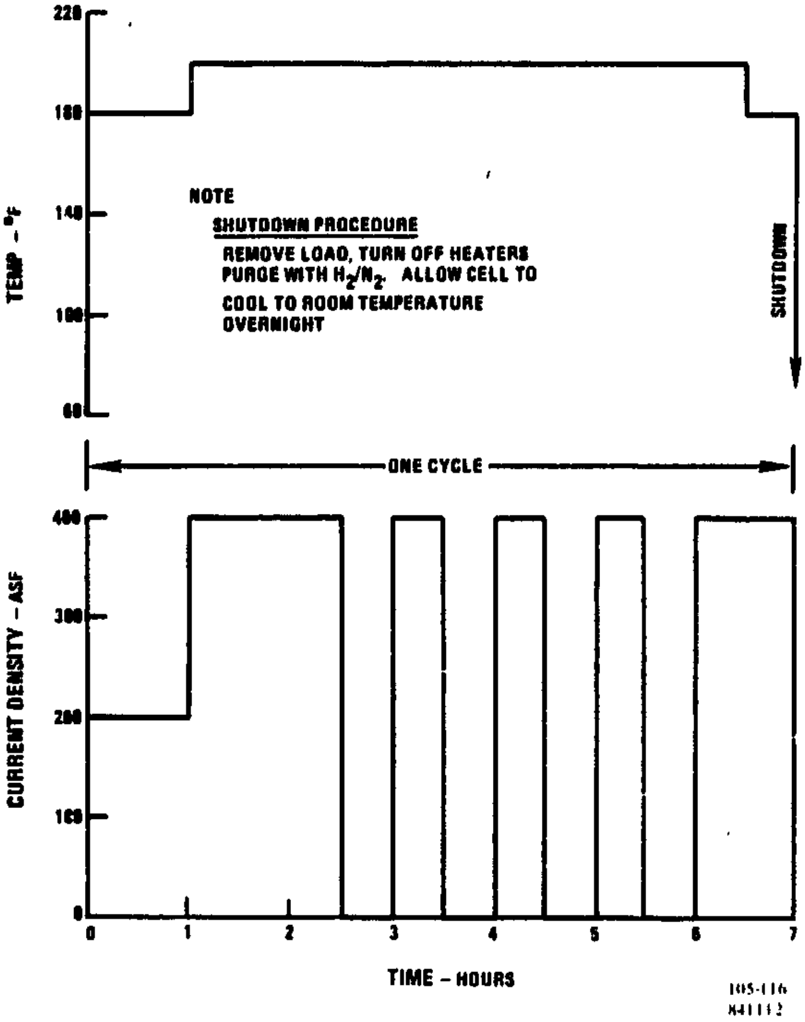

Figure 31. Research Cell (RC-42) Stress Test - Daily Load Profile

RC-42 completed fifteen "stress test" cycles for a total of 2136-hours of testing. Retarding the formation of an electrolyte film on the electrode reactant surface should improve the performance stability of the cell to load and electrolyte volume swings. The "stress test" produces significant changes in the volume of the electrolyte and would be expected to cause the cell to lose performance if the electrode was susceptible to flooding. During the "stress test" period of operation the performance of RC-42 increased 12mV from 0.888V to 0.900V at 200 ASF (215.3 mA/cm^2).

Test results indicate that the selectively wet-proofed anode configuration improves cell tolerance to load and electrolyte volume swings, in fact cell performance increase during the "stress test".

The endurance test of RC-42 was stopped at 2136-load hours to free-up research cell hardware for other planned tests.

RC-43 (Carbon Ribbed Substrate Anode)

RC-43 was the first research cell in the program to incorporate a supported anode catalyst, platinum-on-carbon catalyst layer, on a carbon ribbed substrate. The cell also contained a 20-mil advanced asbestos matrix and a gold-platinum catalyst cathode.

The cell was initially filled with 27% wgt. KOH at room temperature. Upon establishing endurance test conditions, the maximum performance achieved was 0.860V at 200 ASF (215.3 mA/cm^2). Unfortunately a stable performance level could not be achieved and the test was interrupted for an electrolyte refill.

Prior test experience has indicated that carbon ribbed substrates do not "wet" readily with electrolyte and therefore may have insufficient electrolyte with only an initial electrolyte fill.

On returning the cell to the test stand and resuming test cell performance continued to be unacceptable and all further testing was stopped.

RC-43A (Carbon Ribbed Substrate Anode)

The construction of RC-43A was identical to that of RC-43.

Prior to assembling the cell, the anode was filled with electrolyte separately by floating the electrode on a solution of hydrazine-diluted electrolyte. The matrix was filled with 19% wgt. KOH.

The inability to achieve a satisfactory initial performance resulted in interrupting the test after 50-load hours. The performance instability suggested that the cell electrodes were not sufficiently wetted with electrolyte. RC-43A was shutdown and refilled with 35% wgt. KOH electrolyte.

The electrolyte refill did not produce any significant improvement in performance stability. The endurance test was again interrupted after 174-hours of testing.

The previous electrolyte fills should have sufficiently wetted the electrodes with electrolyte. Therefore the approach taken was to improve hydrogen access to the electrode due to the possibility that the anode was starved of fuel. RC-43A was disassembled and a series of holes were drilled through the web between channels of the substrate. The anode was refilled with 27% wgt KOH electrolyte and the cell was reassembled and returned to the test facility.

Upon return to endurance test conditions a stable performance of 0.808V at 200 ASF (215 mA/cm^2) was achieved for 70 load hours. However this performance level was lower than expected and a dilute oxygen diagnostic test was conducted to help ascertain the cause of the low performance. Test results of the dilute gas test are presented on Figure 32.

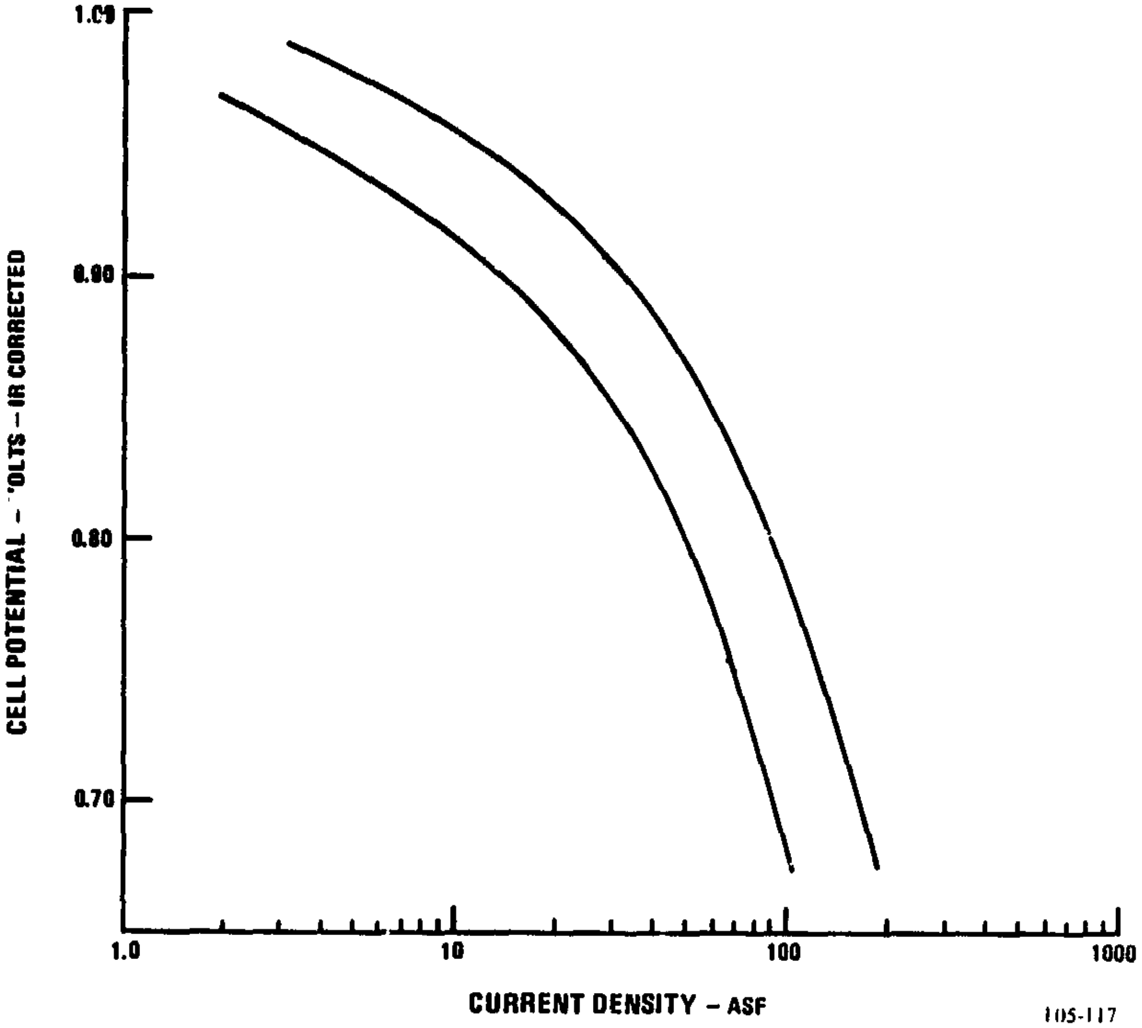

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Figure 32. Research Cell (RC-43A) Dilute Gas Test Results

Dilute gas test results suggests that the majority of the reduced performance was due to cathode polarization. However, when the cell endurance test was stopped at 338 load-hours, samples of the cathode were half-cell tested showing no sign of reduced cathode performance. The half-cell tests revealed a normal cathode performance.

A likely explanation for the reduced performance of RC-43A is that excess electrolyte was being forced into the cathode resulting in thick electrolyte films in catalyst pores of the cathode.

Assuming that this hypothesis is correct, there are several approaches which are available to overcome the deficiency.

- Treat the ribbed substrate chemically or physically to enhance its wettability and increase propensity to wick up and retain electrolyte. As an approach, introduce a wettable powder into the pores or alter the surface condition of the graphite fibers by oxidation or coating with a wettable film.
- Reduce the Teflon® content of the anode catalyst layer.

The approach taken by Power Systems Division was to introduce a wettable material into the carbon structure.

<u>RC-44 (Carbon Ribbed Substrate Anode)</u>

As an approach to improving the electrolyte-wetting characteristics of the substrate, the anode ribbed substrate was impregnated with 2-3 mg/cm^2 of graphite prior to depositing the catalyst layer. The resultant anode, a gold-platinum catalyst cathode and 20-mil (.5mm) reconstituted asbestos matrix were assembled into research cell RC-44.

The maximum performance achieved on RC-44 was 0.856V at 200 ASF (215.3 mA/cm^2). In the subsequent 84-hours of testing, which included an electrolyte refill, cell voltage continued to fall off. A total of 295-hours of testing was completed on RC-44 and the test was stopped because of the inability to achieve a satisfactory performance level.

Prior to stopping the endurance test, a series of reactant cross pressure tests were conducted to help identify the electrode responsible for the low performance. The anode and cathode reactant cavity were individually subjected to 5 psi (3.4 N/cm^2) over pressures. Increasing the reactant pressure on the cathode had no effect on cell voltage; however, increasing the reactant pressure on the anode resulted in a 45 mV increase at 200 ASF (215.3 mA/cm^2) in cell performance. Test results indicate that the anode was operating at a flooded condition.

RC-45 (Carbon Ribbed Substrate Anode)

Research cell RC-45 was constructed with a platinum-on-carbon catalyst anode, deposited on a carbon ribbed substrate which had been selectively wet proofed with Teflon® layer at the catalyst substrate interface. There was no special treatment of the substrate to promote electrolyte wetting. However the individual cell components, matrix and electrodes, were filled with electrolyte prior to cell assembly and the assembled cell was flush filled with 23% wgt. KOH before the start of the endurance test.

The peak performance of RC-45 was 0.860V at 200 ASF (215.3 mA/cm^2). A dilute oxygen test performed at 32-load hours, Figure 33, revealed that the cell was experiencing some reactant cross leakage or a small internal short. The performance anomaly forced the endurance test to be stopped after 200-hours of testing.

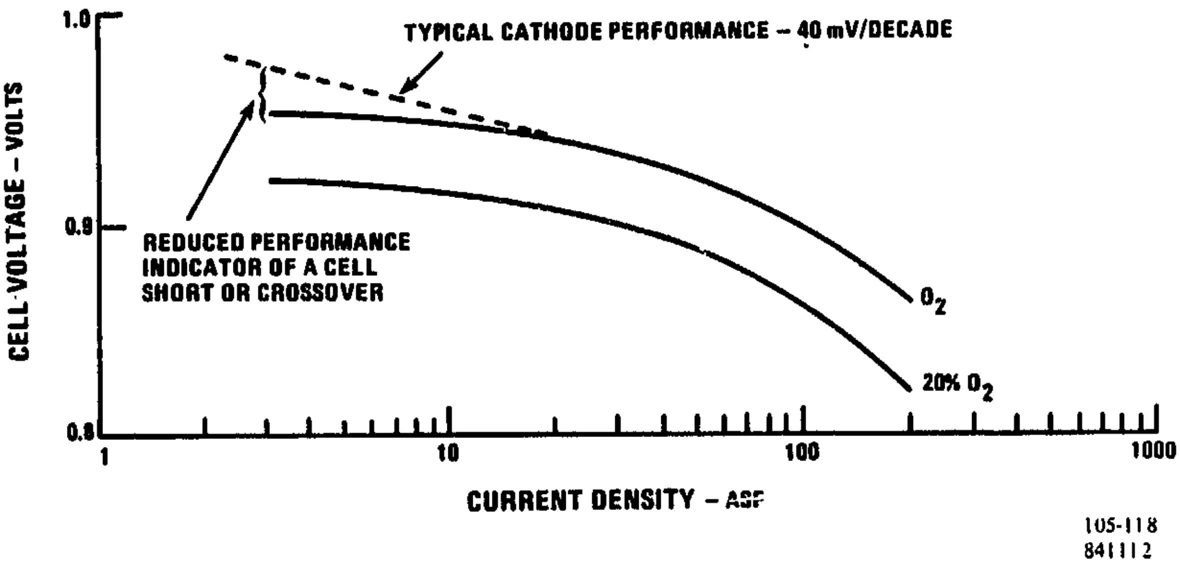

Figure 33. Research Cell (RC-45) Dilute Oxygen Test

RC-46 (Carbon Ribbed Substrate)

This cell was constructed with a platinum-on-carbon anode catalyst on a nickel-impregnated carbon ribbed substrate, a gold-platinum cathode catalyst layer on a gold-plated nickel screen and a 20-mil (.51mm) thick reconstituted asbestos matrix. The cathode and matrix of RC-46 were unitized into a fiberglass/epoxy edge frame.

The performance characteristics of RC-46 with time matched past test experience of research cells fabricated with ribbed-substrate anodes, that is, the cells lose performance from the initial level, however a majority of the performance is restored following dilute-oxygen tests or by electrolyte filling. Test results from dilute oxygen tests reveal only a small change in the performance of the cathode as shown in Figure 34. The majority of the reduced performance occurs as an increase in anode polarization, as shown on Figure 35.

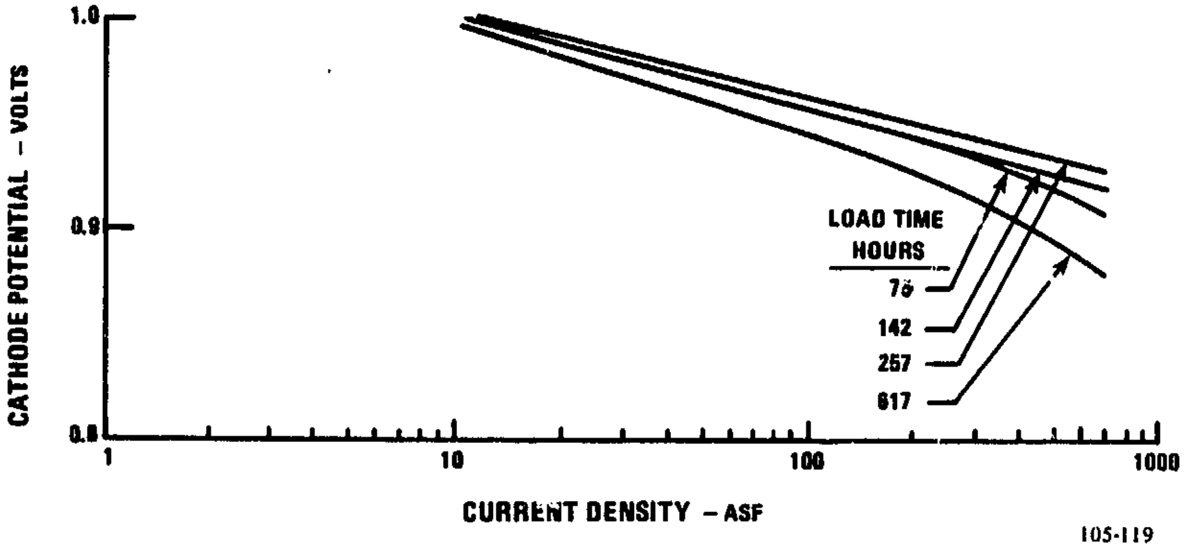

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Figure 34. Research Cell (RC-46) Dilute Oxygen Test, Cathode Potential

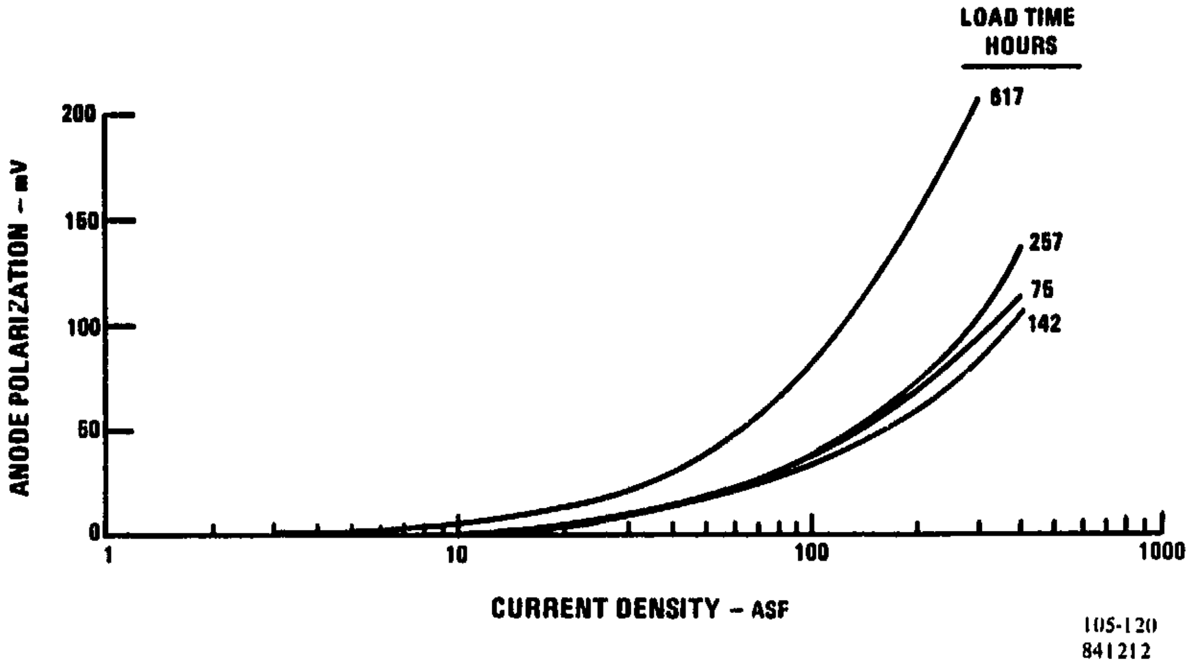


Figure 35. Research Cell (RC-46) Dilute Oxygen Test, Anode Polarization

The endurance test was stopped after 648 load hours because of the inability to maintain an acceptable performance level.

The nickel impregnation of the carbon ribbed substrate resulted in a structure with improved electrolyte wetting and improved performance stability.

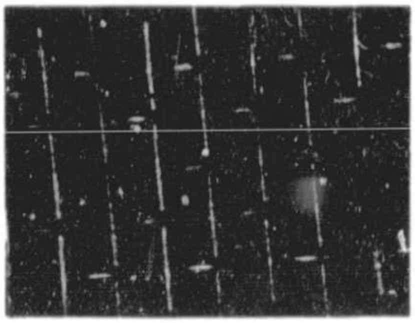
RC-47 (Carbon Ribbed Substrate Anode)

The construction of RC-47 was identical to that of research cell RC-46 except that the carbon ribbed substrate had a selectively wet-proofed layer at the catalyst-substrate interface.

The inability to achieve a satisfactory performance level forced stopping the endurance test after 280-hours of testing.

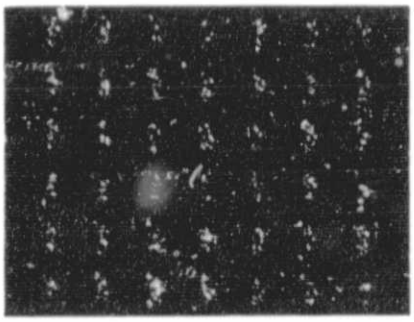
RC-48 (Photo-Fabricated Nickel Foil Substrate Anode)

This research cell incorporates the first anode under the program on a perforated, photo-fabricated nickel foil. The nickel foil was a commercially available material. The sample foil purchased measured 12 x 18 x 0.003 in. (30.5 x 45.7 x .008 cm) and had a hole pattern of 0.005 in (0.013 cm) diameter circular openings with a 0.005 in. (0.013 cm) web, arranged in a square array. In order to protect the substrate during operation in the cell, the photo-fabricated foil was gold plated. A photomicrograph of the gold-plated foil and of a standard gold-plated nickel screen, at the same magnification, is shown in Figure 36.



STANDARD GOLD-PLATED
Ni SCREEN

(W-5106)



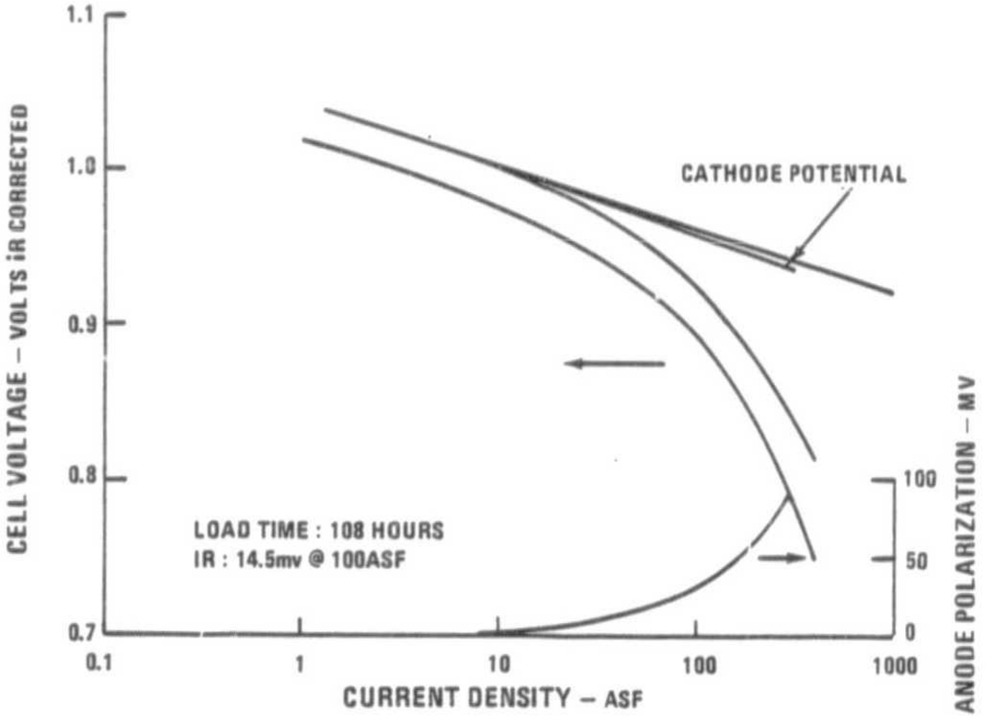
GOLD-PLATED NICKEL
PHOTO-FABRICATED FOIL

(W-5107)

Figure 36. Electrode Substrate Comparison

RC-48 was constructed with a supported platinum catalyst anode, gold-platinum catalyst cathode and a 20-mil (.5mm) thick advanced asbestos matrix. A 100-mil (2.5mm) porous nickel electrolyte reservoir plate was installed on the gas side of the anode.

The initial cell performance was 0.848V at 200 ASF (215.3 mA/cm²). A dilute-oxygen test, shown on Figure 37 at 108 hours showed that the cathode was essentially activation controlled and that the deviation of the cell performance from the Tafel curve was attributable to the anode.



105-98

Figure 37. Research Cell (RC-48) Dilute Oxygen Test Results

The performance slowly fell-off with time and the endurance test was stopped at 300-load hours due to low performance. The final cell performance was 0.821V at 200 ASF (215.3 mA/cm²) and 200°F (93.3°C).

When fabricating the anode catalyst layer on the perforated-nickel foil substrate, there appeared to be little penetration of the catalyst into the holes resulting in the catalyst layer being confined to one side of the substrate. In RC-48, the anode was operated with the catalyst layer adjacent to the matrix. With this cell configuration, electrolyte transfer between the catalyst layer and electrolyte reservoir plate may have been restricted.

RC-48A (Photo-Fabricated Nickel Foil Substrate Anode)

The cell construction of RC-48A was identical to that of research cell RC-48. In the assembly of the cell, however the anode catalyst layer was next to the electrolyte reservoir plate.

Reversing the anode catalyst layer did not improve performance. The performance of RC-48A was lower than RC-48. The endurance test was stopped after 37 hours of testing.

Test results from RC-48 and RC-48A indicate that the configuration of the perforated nickel foil is far from ideal for use as an electrode substrate. Geometric calculations showed that the open area was less than fifteen percent which could restrict the transport of both electrolyte and reactant gas. Further calculations showed that by increasing the size of the holes and arranging them in a close-packed array, a porosity of at least fifty percent could be obtained. An order for the revised configuration nickel-foil was placed to be evaluated in further research cells.

RC-49 (Carbon Ribbed Substrate Anode)

Impregnation of the ribbed carbon substrate with nickel oxide, formed from thermal decomposition of an aqueous nitrate wash coat, has been shown to improve the wettability of the substrate. This procedure was employed in preparing the anode ribbed carbon substrate in RC-49. In addition, in order to improve the gas supply to the anode catalyst layer, a series of holes was drilled in the web of the substrate.

The endurance test of RC-49 was begun in the laboratory and attained a peak performance of 0.875V at 200 ASF (215.3 mA/cm²) after 16 load-hours. After 112-load hours of testing cell voltage was 0.840V. At this point in the test, the load was removed and the anode was purged with nitrogen and then exposed to oxygen for two-minutes. On resumption of the test, cell performance was 0.864V. The performance recovery suggests that the lower cell voltage with carbon ribbed substrates may be due to the accumulation of oxidizable impurities on the catalyst surface. Also, raising the anode-to-oxygen potentials may have caused the catalyst layer to take-up more electrolyte and increase working area.

The performance history of RC-49 is presented on Figure 38.

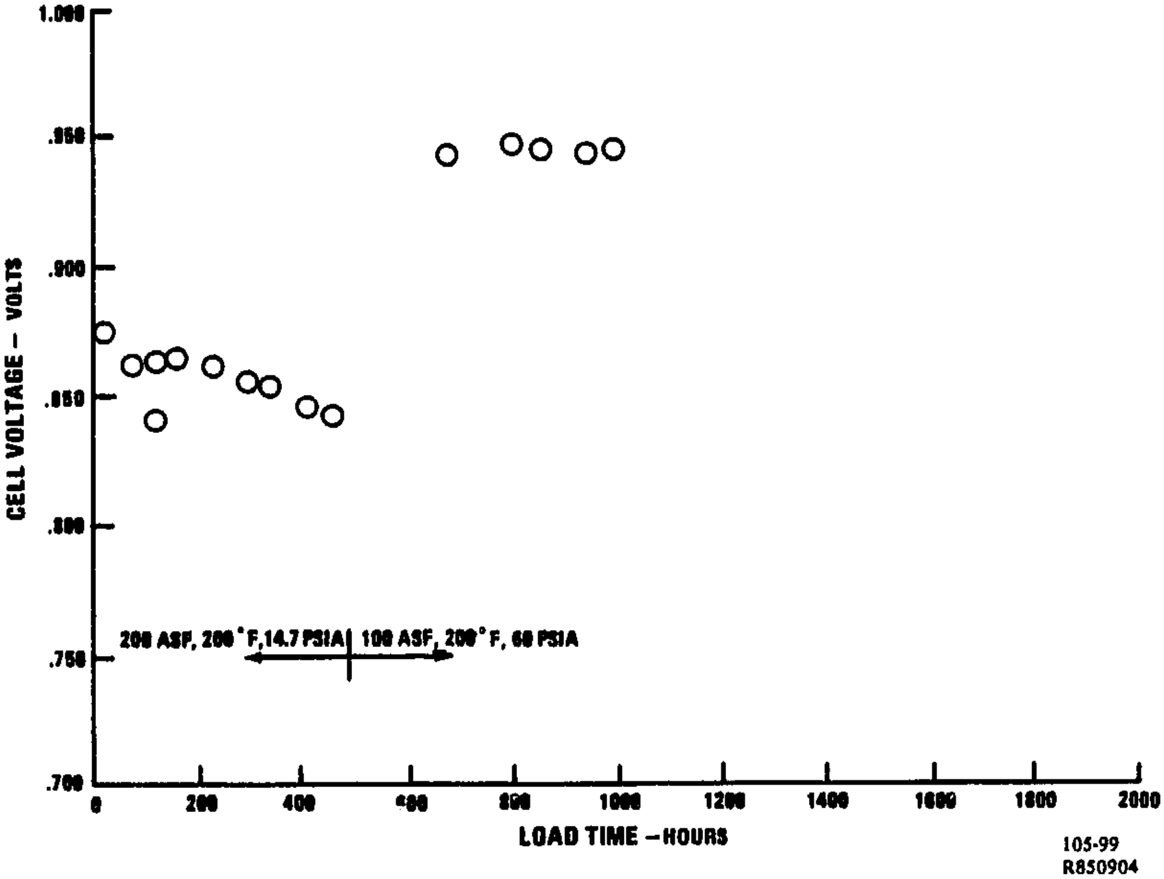


Figure 38. Research Cell (RC-49) Endurance Test - Nickel Impregnated Carbon Ribbed Substrate Anode

At 457-load hours the endurance test of RC-49 was interrupted for the Christmas holiday. At this point the cell was flush-filled with 23% wgt. KOH electrolyte.

Instead of returning the cell to the laboratory test facility, which can only operate at atmospheric pressure, the cell was transferred to a commercial endurance test bench, where the cell could be operated at elevated reactant pressure. Very good performance has been seen on alkaline fuel cells with carbon electrolyte reservoir plates when operated at elevated reactant pressure (reference NASA CR-165417). The voltage fall-off with time experienced at ambient pressure is attributed to a redistribution of the stored electrolyte which leads to increased anode polarization.

The initial performance at 200 ASF (215.3 mA/cm^2) 60 psia (41.4 N/cm^2), and 200°F (93.3°C) upon achieving stabilized operating conditions was 0.942V. During the 763-hours of testing at 60 psia (41.4 N/cm^2) for a total of 1250 total test hours, there was no loss in cell performance. The performance at the completion of the planned endurance test was 0.942V. The stable performance of RC-49 during the 60 psia (41.4 N/cm^2) reactant pressure demonstrates the suitability of the carbon ribbed substrate anode for use in the alkaline fuel cell.

RC-50 (Carbon Ribbed Substrate Anode)

Research cell RC-50 was fabricated with a platinum-palladium catalyst layer on a nickel oxide impregnated, carbon ribbed substrate with holes drilled through the web to the catalyst layer. The substrate was identical to that employed in RC-49. The platinum-palladium catalyst layer was the same as that used in the Space Shuttle Orbiter fuel cell powerplant.

In contrast to earlier performance evaluation tests of carbon ribbed-substrate anodes at atmospheric pressure, the performance stability of RC-50 at ambient pressure in the laboratory test stand was very good. The initial performance was 0.896V at 200 ASF (215.3 mA/cm^2) and at the completion of the test at 1357 load hours, having exceeded the 1000-hour program test goal, the cell voltage was 0.878. The performance history of RC-50 is shown on Figure 39.

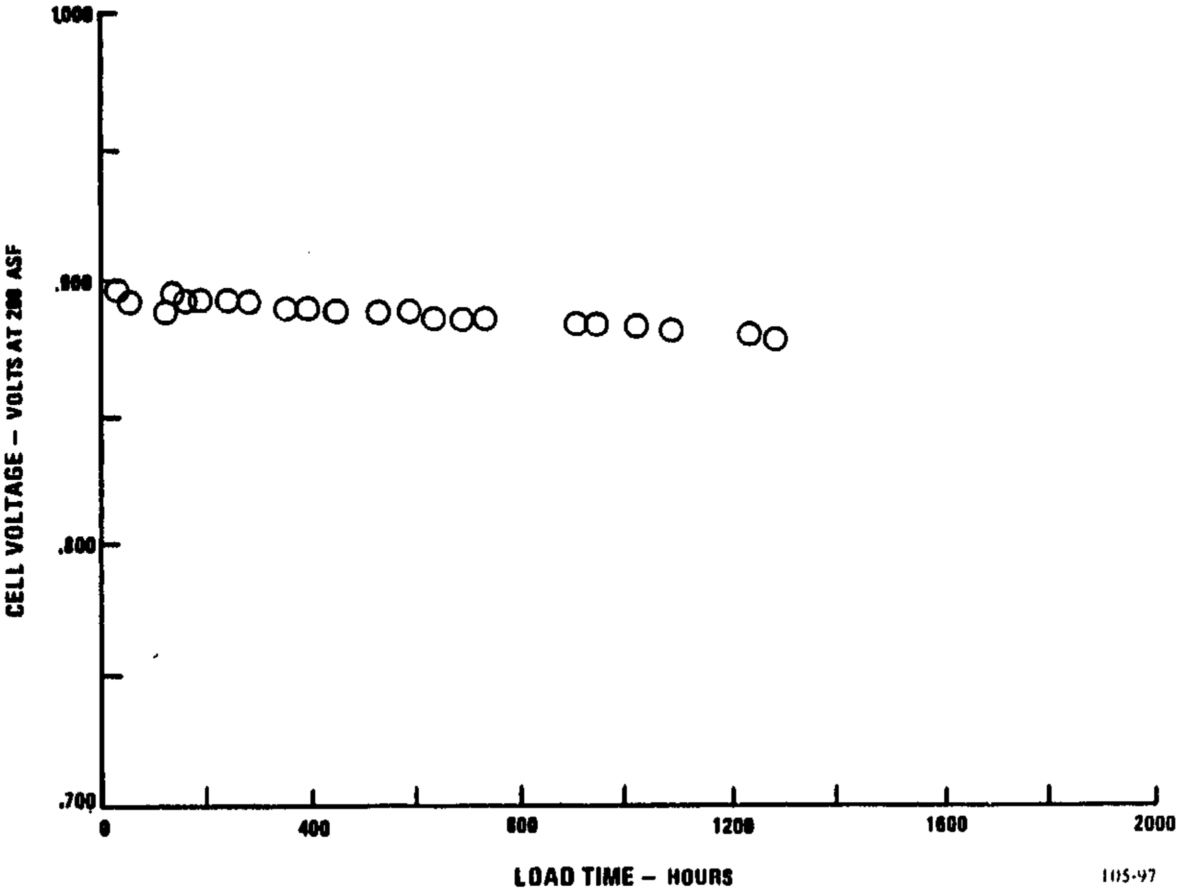


Figure 39. Research Cell (RC-50) Endurance Test
Nickel-Treated Carbon Ribbed Substrate Anode

The stable performance demonstrated during the test is an indication that the anode polarization losses experienced with previous platinum-on-carbon catalyst layers on graphitized ribbed substrates tested at ambient pressure have occurred at the catalyst layer/substrate interface.

RC-51 (Carbon Paper Substrate Anode)

RC-51 was built for a brief exploratory test to determine if there would be any change in anode behavior if the platinum-on-carbon catalyst layer was applied to a thin non-ribbed carbon paper and was run in conjunction with a nickel-treated ribbed carbon electrolyte reservoir plate, in effect employing two carbon components to perform the separate functions of supporting the catalyst layer and accommodating changes in electrolyte volume.

Two variations were tested. The first with the electrolyte reservoir plate ribs facing away from the anode and a second with the ribs facing the anode. The latter configuration appears to be the better arrangement, but in neither case was the performance acceptable.

A total of 166-hours of testing at 200°F (93.3°F), 200 ASF (215.3 mA/cm^2) and atmospheric pressure completed.

RC-52 (Bonded Potassium Titanate Matrix)

A 95% wgt potassium titanate/5% wgt Teflon® bonded was incorporated into RC-52.

A total of 2225 hours of testing at 200 ASF (215.3 mA/cm^2), 200°F (93.3°C) and atmospheric pressure. The test was stopped in order to relocate the laboratory test facility.

The performance history of RC-52 is presented on Figure 40. The initial cell voltage was 0.909V and after 2225 hours cell performance was 0.886V.

The performance characteristics with time of RC-52 was similar to that experienced by RC-54 and RC-60 which contained identical electrodes. Test data from dilute oxygen diagnostic tests conducted on RC-54 and RC-60, which are discussed later, indicates that the voltage fall-off with time was principally due to a decrease in cathode activity and an increase in anode polarization.

Test results from the endurance test of RC-52 indicates that the Teflon®-bonded potassium titanate matrix is stable in the alkaline fuel cell environment. In addition, the bonded potassium titanate matrix cells have consistently shown higher performance than standard asbestos cells because of lower internal resistance (iR), 8 mV @ 100 ASF (107.6 mA/cm^2) for potassium titanate matrix cells compared to 11 mV for asbestos matrix research cells.

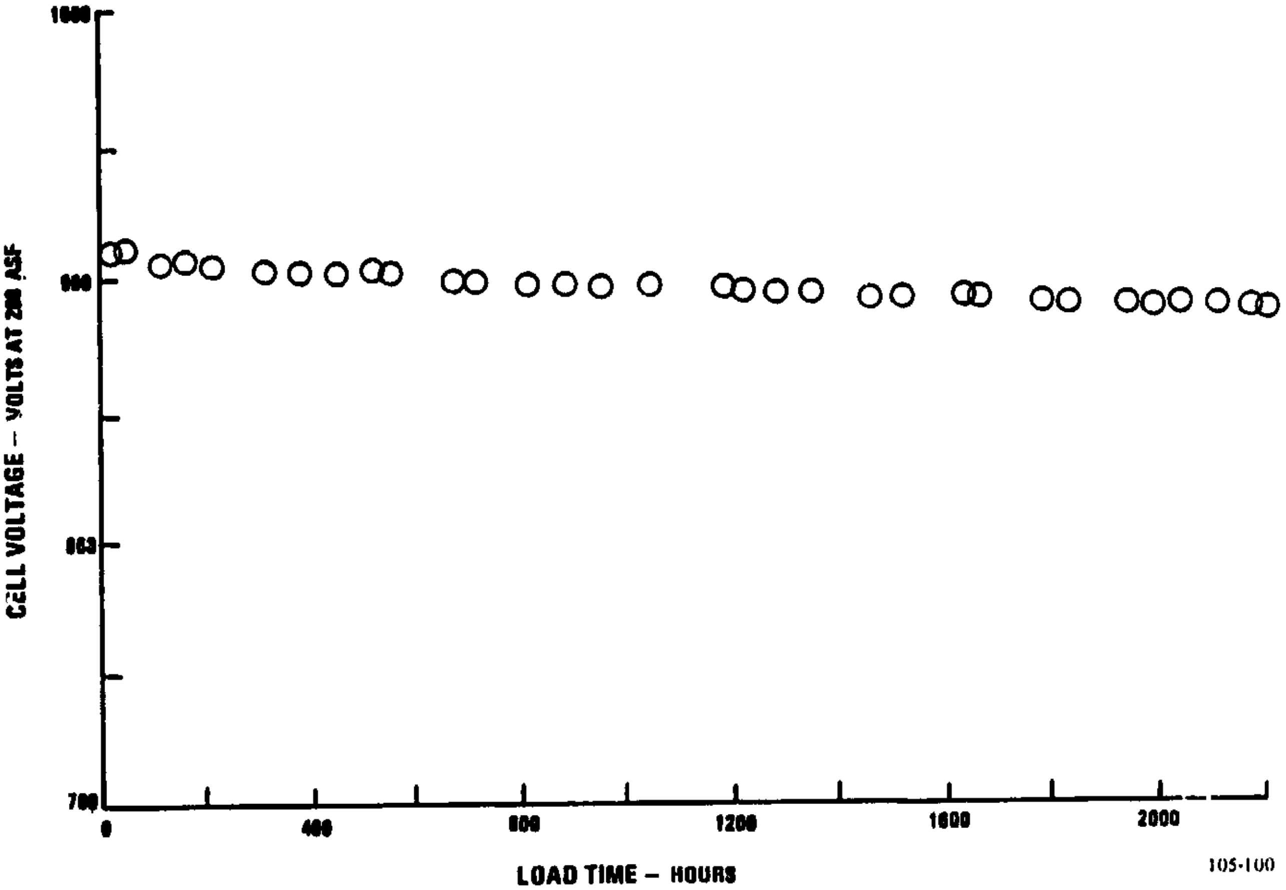


Figure 40. Research Cell (RC-52) Endurance Test - Bonded Potassium Titanate Matrix

RC-53 (Ribbed Nickel Substrate Cathode)

Research cell RC-53 was the first cell during the program to incorporate a ribbed nickel substrate. The substrate was machined from a nickel felt material and subsequently coated with nickel-cobalt spinel. A 20 mg/cm^2 loaded gold platinum catalyst layer was deposited on the substrate.

The endurance test was stopped after 174 hours because an acceptable performance level could not be attained.

The low performance experienced on RC-53 appears to be typical of the performance experienced on past research cells incorporating cathode catalyst layers supported on nickel, or nickel coated with a protective oxide film. The low performance is attributed to a low electrical conductivity at the catalyst/substrate interface.

RC-54 (Bonded Potassium Titanate Matrix)

A 4% wgt butyl rubber bonded/96% wgt potassium titanate matrix was incorporated into research cell RC-54.

A total of 1660 hours of testing exceeding the 1000 hours program test objective was completed. The final cell performance was 0.889 volts at 200 ASF (215.3 mA/cm²) compared to a initial voltage of 0.913 volts. The performance history of RC-54 is presented on Figure 41.

Endurance testing of RC-54 was stopped in order to relocate the in-house laboratory test facility.

The performance characteristics of RC-54 were similar to RC-52, in fact, at the shutdown of RC-54 at 1660-hours, the cell voltage was identical to RC-52 at the same load time. Test results indicate that both the butyl rubber binder and Teflon® binder are stable for 1660 hours in a 200°F (93.3°C) fuel cell. Additional testing will be required to verify the long-term stability of these binders in the fuel cell environment. However, from the stand point of ease of manufacture and mechanical strength the butyl rubber bonded potassium titanate matrix may be preferable to the Teflon®-bonded Matrix.

RC-55 (Ribbed Nickel Substrate Cathode)

Research cell RC-55 was the first cell to operate successfully with a ribbed substrate cathode. The substrate was machined from a nickel felt material, identical to the substrate employed in RC-53, except in RC-55 the substrate was plated with electroless gold prior to applying the cathode catalyst layer. The catalyst consisted of gold-platinum with a loading of 20 mg/cm².

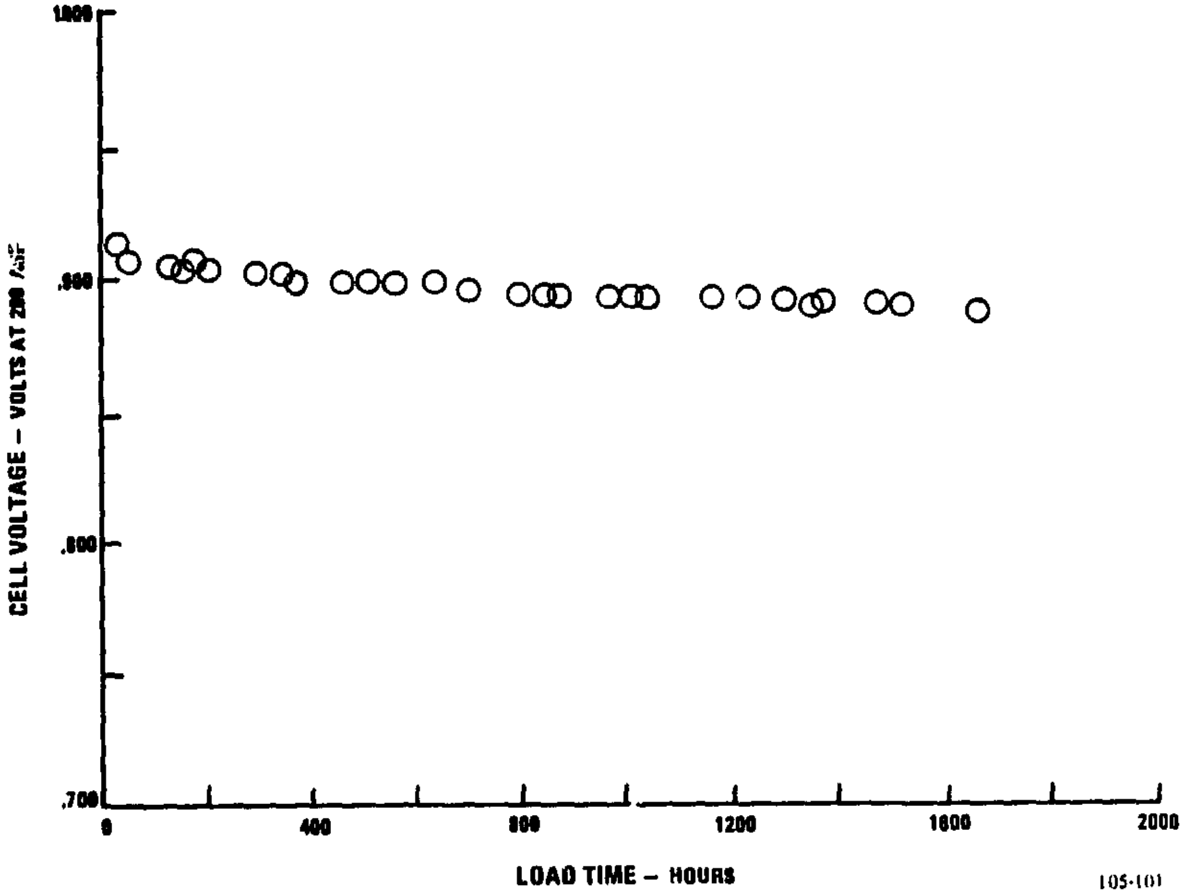


Figure 41. Research Cell RC-54 Endurance Test - Butyl Rubber Bonded Potassium Titanate Matrix

The cell completed at total of 1007 hours of testing. The test was stopped upon completion of the planned 1000 hour test.

The performance history of RC-55 is presented on Figure 42. The initial cell voltage was 0.921V at 200 ASF (215.3 mA/cm^2) and at the completion of the test, performance was 0.896 volts. The voltage fall-off with time on RC-55 was similar to previous laboratory research cells incorporation of gold-plated nickel screen substrate cathodes. There was no evidence in the test data that the ribbed nickel felt substrate contributed to the voltage fall-off. In addition the substrate appeared to be stable at cathode potentials as cell iR remained constant at 11 mV at 100 ASF (107.6 mA/cm²). An 11 mV iR is consistent with past test experience.

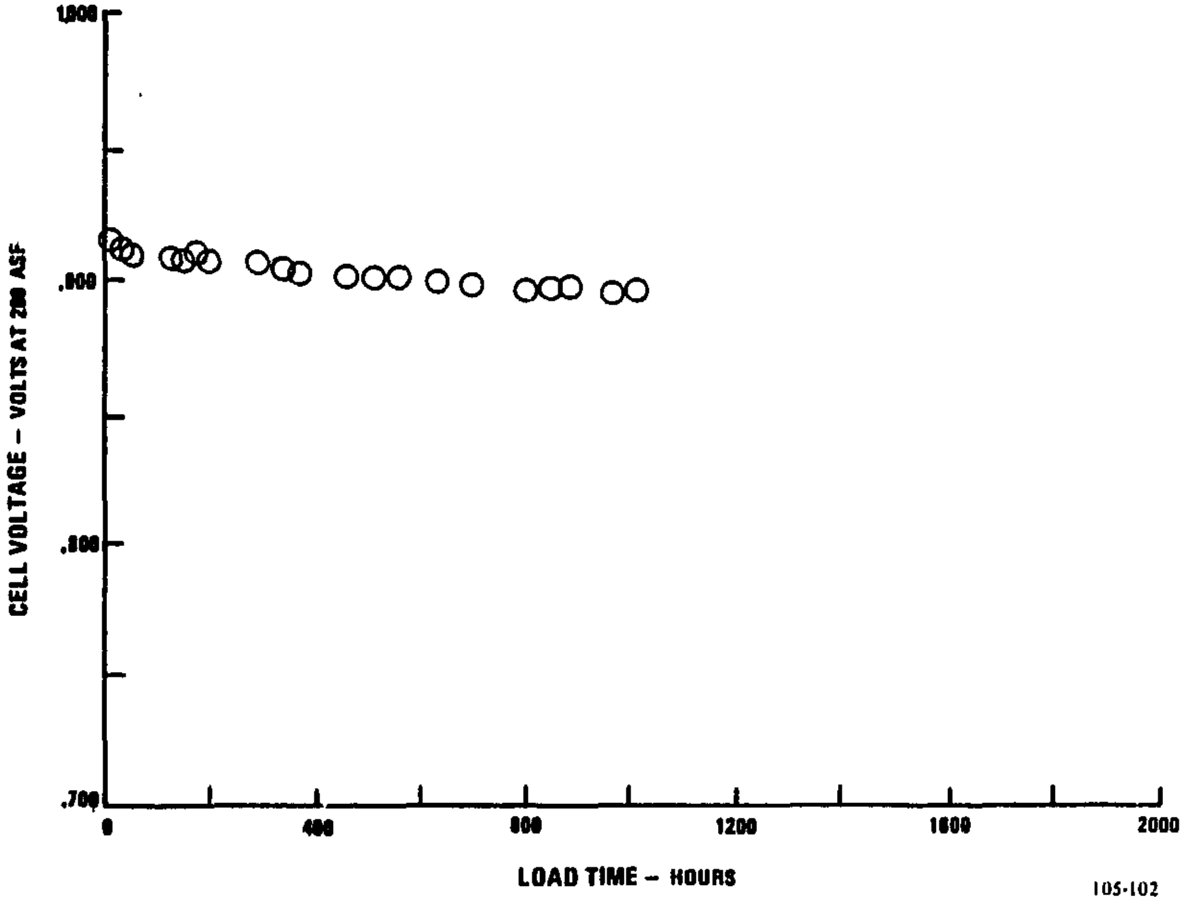


Figure 42. Research Cell RC-55 Endurance Test - Gold-Plated Ribbed Nickel Felt Substrate Cathode

RC-55 was initially flush filled with electrolyte, it is more than likely that electrolyte was stored not only in the porous nickel electrolyte reservoir plate against the anode but also in the porous ribbed nickel felt substrate cathode. This cell configuration should be very tolerant to reactant pressure imbalances which can occur during the operation of the fuel cell. However, no specific tests were conducted to verify this capability. In addition, the increased volume of stored electrolyte will help to extend cell life by reducing the impact of electrolyte carbonation and the associated impact upon cell performance.

RC-56 (Ribbed Nickel Substrate Cathode)

In a brief exploratory test, RC-56 was built to determine if there was sufficient gold on the gold-plated, ribbed, nickel felt substrate to support the cathode reaction in the absence of a separate catalyst layer.

Unfortunately an acceptable performance level could not be established and the test was stopped after 2-load hours.

RC-57 (Carbon Ribbed Substrate Anode)

RC-57 was a composite cell incorporating a platinum-palladium anode catalyst layer on a nickel treated ribbed carbon substrate and a gold-platinum cathode catalyst layer on a ribbed gold-plated nickel felt substrate.

The performance history of RC-57 is presented on Figure 43. The initial cell performance was very good, 0.907 volts at 200 ASF (215.3 mA/cm^2) and upon completion of the test at 500-load hours, cell voltage was 0.889 volts. The voltage fall-off with time was similar to that experienced on RC-50 which had an identical anode, but a gold-platinum screen substrate cathode.

The performance levels of RC-50 and RC-57 were identical at 500-hours when RC-57 was shutdown. RC-57 was shutdown to make hardware available for other planned research cell tests under the program.

Test results indicate that the ribbed nickel felt substrate cathode is stable at cathode potentials, even with stored electrolyte, and does not contribute any additional diffusional loss when compared to the standard nickel screen substrate.

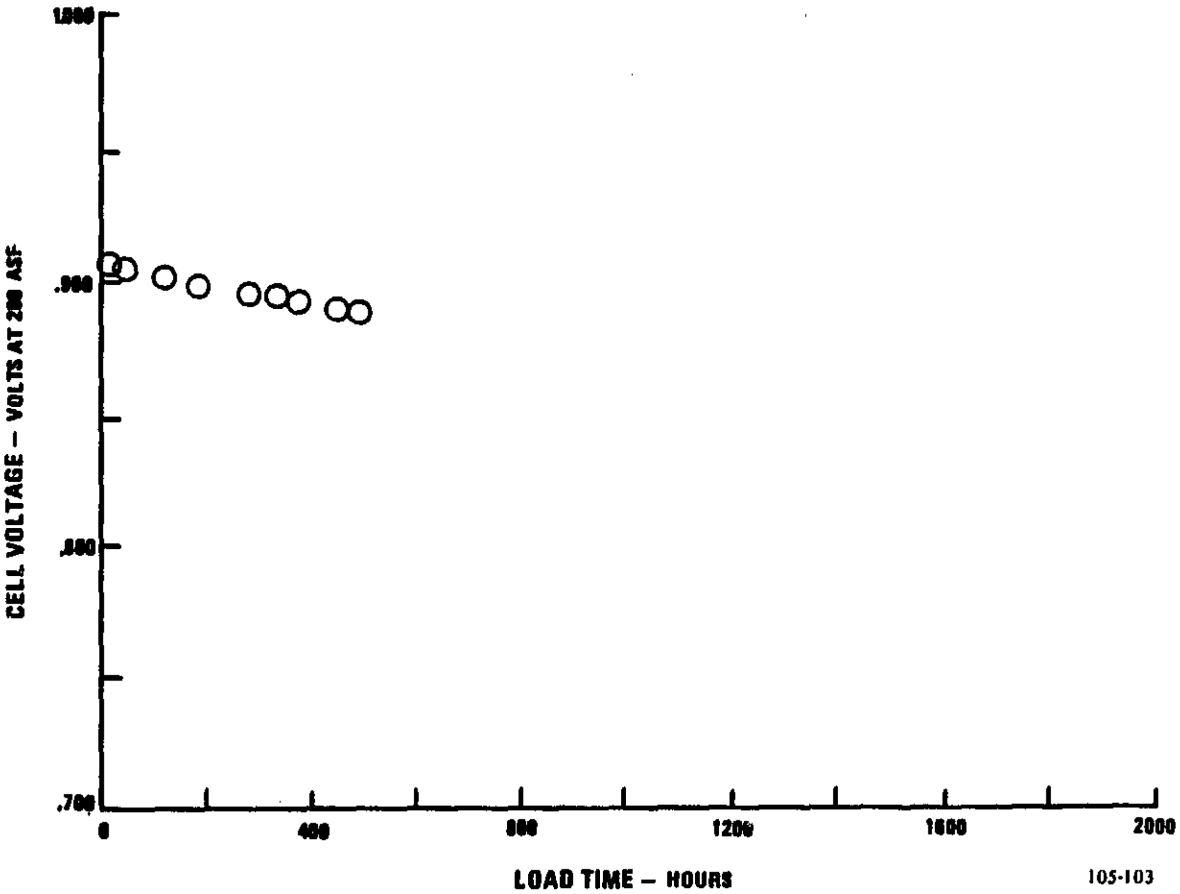


Figure 43. Research Cell RC-57, Endurance Test - Carbon Ribbed Substrate Anode

RC-58 (Gold Plated Photo-Fabricated Nickel Foil Substrate Cathode)

RC-58 was constructed with a standard production platinum-palladium catalyst anode and a gold-platinum cathode catalyst layer on a gold-plated nickel photo-fabricated foil. Other details of cell construction is shown in Table XIII, Section V.

The initial cell performance was very good, 0.902 volts at 200 ASF (215.3 mA/cm²). in 938 hours of testing RC-58 had no reduction in cell voltage. The final cell performance was 0.903 volts. The test was stopped in order to relocate the laboratory test facility.

Half-cell performance tests have revealed that the long-term performance stability of RC-58 can be attributed to the gold-platinum catalyst employed in fabricating the cathode. Half-cell and laboratory research cell tests have shown that 24 mil (.61mm) diameter hole nickel foil configuration is a suitable substitute for the 100-mesh screen currently employed in making cathodes.

RC-59 (Photo-Fabricated Nickel Foil Substrate Anode)

This cell was constructed with a cathode similar to research cell RC-58 and a platinum-on-carbon anode catalyst layer on a photo-fabricated nickel foil configuration substrate.

The test was stopped after 160-hours of testing because of the inability to achieve an acceptable performance level. The inadequate performance level was attributed to the anode because of the very good performance stability experienced with an identical cathode in RC-58.

RC-60 (Bonded Potassium Titanate Matrix)

The construction of RC-60 was similar to research cell RC-54, that is, RC-60 contains a standard production anode and cathode, a 51-mil (1.3mm) thick electrolyte reservoir plate and a 20-mil (.5mm) 4% wgt butyl rubber bonded 96% wgt potassium titanate matrix.

The purpose of the test was to evaluate the performance characteristics of the matrix in a fuel cell operating at 250°F (121.1°C).

Research cell RC-60 was operated for the first 114-hours of testing at a cell temperature of 200°F (93.3°C). The cell performance during this period was 0.905 volts at 200 ASF (215 mA/cm^2).

Prior to commencing the planned 250°F (121.1°C) endurance test, performance data was obtained at cell temperatures of 200°F (93.3°C), 226°F (107.8°C) and 250°F (121.1°C). Figure 44 shows the effect of cell temperature on RC-60 cell performance. Increasing the cell operating temperature from 200°F (93.3°C) to 250°F (121.1°C) resulted in a 33 mV at 200 ASF (215.3 mA/cm²) increase in cell performance which is consistent with past test experience.

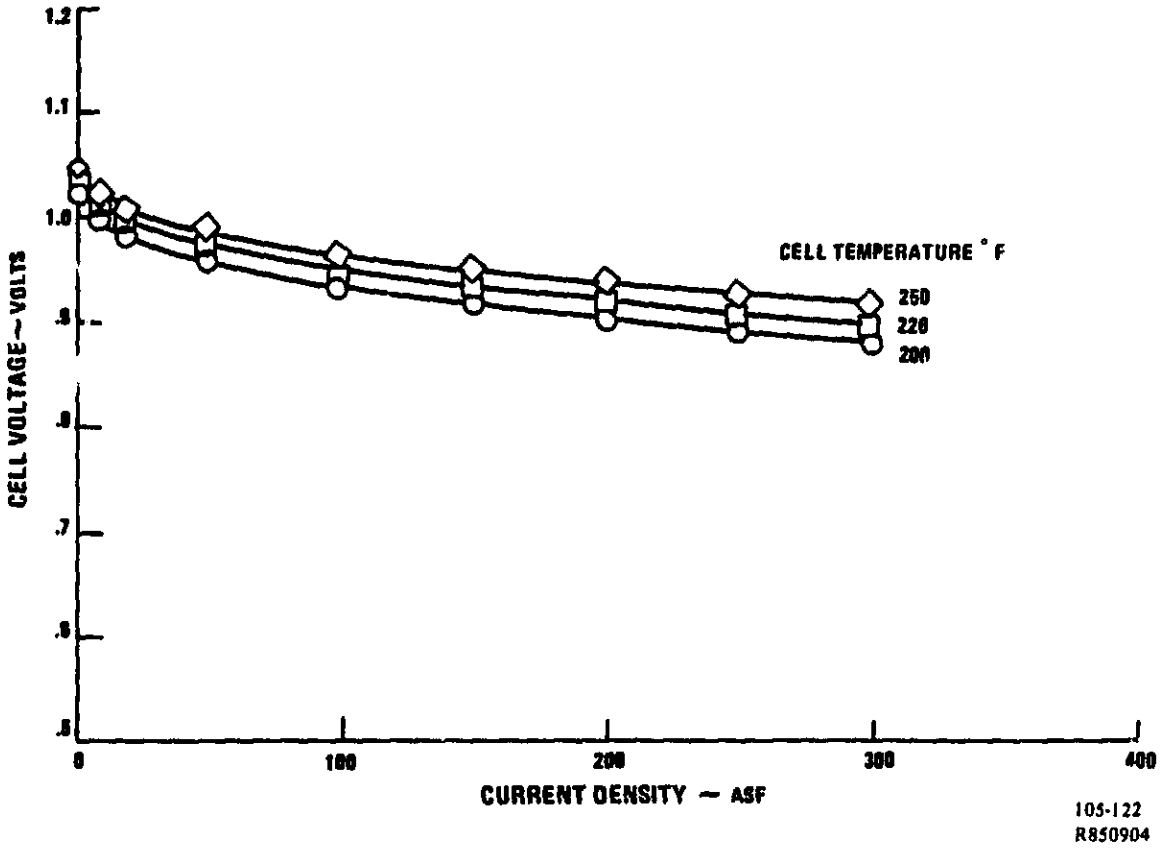


Figure 44. Research Cell RC-60 Performance Calibration (96% Potassium Titanate/4% Butyl Rubber)

Upon completion of the performance calibration the endurance test of RC-60 at a cell temperature of 250°F (121.1°C) was begun. The performance history of RC-60 is presented on Figure 45. The initial performance at 250°F (121.1°C) was 0.949 volts at 200 ASF (215.3 mA/cm^2). Research cell RC-60 was shutdown after completing the program test goal of 1000-hours of operation. RC-60 completed a total of 1125 hours of testing including 1015 hours of testing at 250°F (121.1°C), 200 ASF (215.3 mA/cm^2) and one atmosphere.

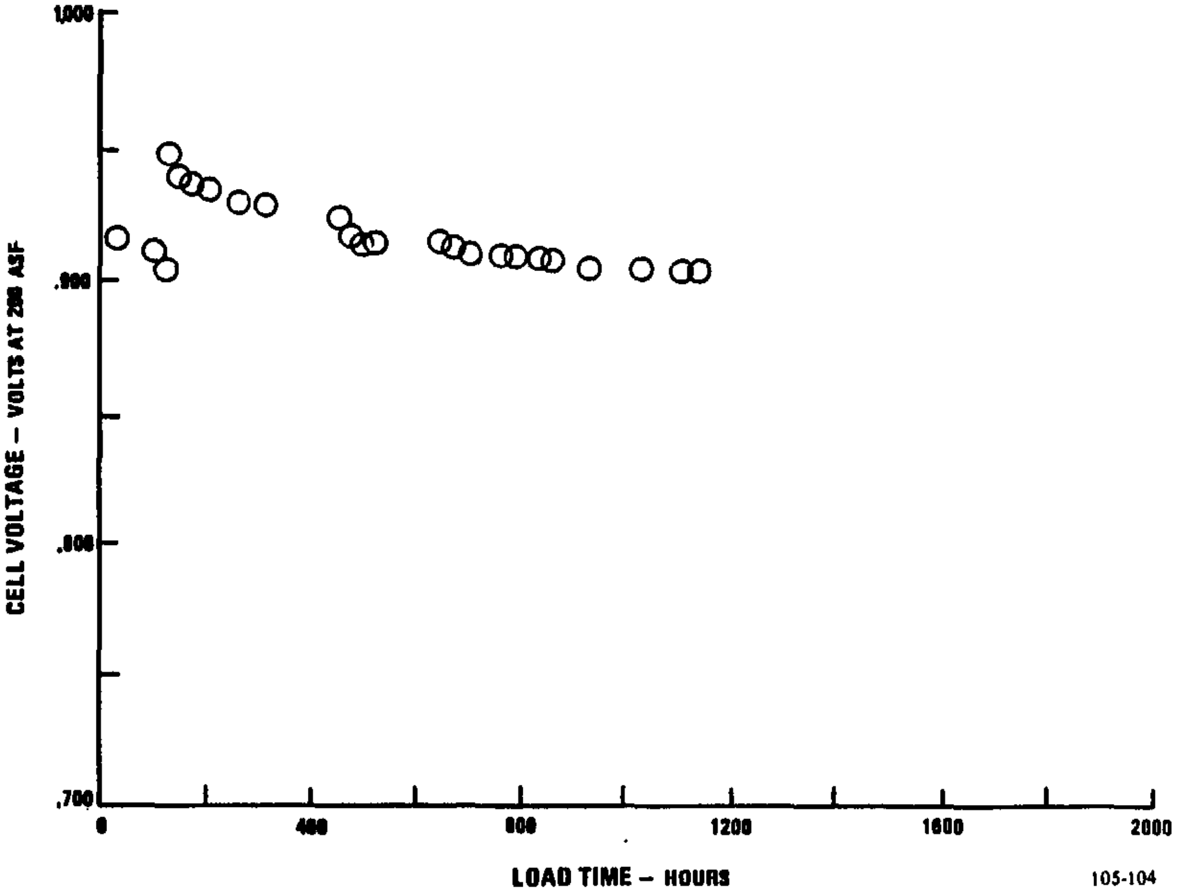


Figure 45. Research Cell RC-60 Endurance Test - 96% Potassium Titanate/4% Butyl Rubber

Dilute oxygen diagnostic test data indicated that the 45mV at 200 ASF (215.3 mA/cm^2) reduction in performance during 250°F (121.1°C) operation was made up of the following individual losses:

• Decrease in cathode activity	11 mV
• Increase in anode polarization	28 mV
• Increase in cell iR	6 mV

The 11 mV decrease in cathode activity is consistent with past research cell test experience on gold-platinum catalyst cathodes. The increase in internal resistance (iR) and a portion of the anode polarization occurred following a lengthy shutdown when the test stand was relocated.

RC-61 (Bonded Potassium Titanate Matrix)

Research Cell RC-61 was constructed with a 95% wgt potassium titanate/5% wgt Teflon® bonded matrix.

The performance history of RC-61 is shown on Figure 46. The cell successfully completed 1135-hours of testing, exceeding the 1000-hour test goal at 250°F (121.1°C), 200 ASF (215.3mA/cm^2) and one atmosphere.

The cell performance of RC-60 and RC-61 after 1000-hours of testing at 250°F (121.1°C) was very similar. The reduction in cathode activity was consistent with previous test experience on gold-platinum cathodes operating at 250°F (121.1°C). The performance characteristics of RC-61 and RC-60 are summarized in Table XVI.

The anode in RC-60 (butyl rubber bonded matrix) exhibited the greater change in polarization compared to the anode in RC-61 (Teflon®-bonded matrix). These test results along with weight-loss data measured in laboratory corrosion tests confirm, as suspected, that the butyl rubber binder is less stable than the Teflon® in aqueous potassium hydroxide at 250°F (121.1°C).

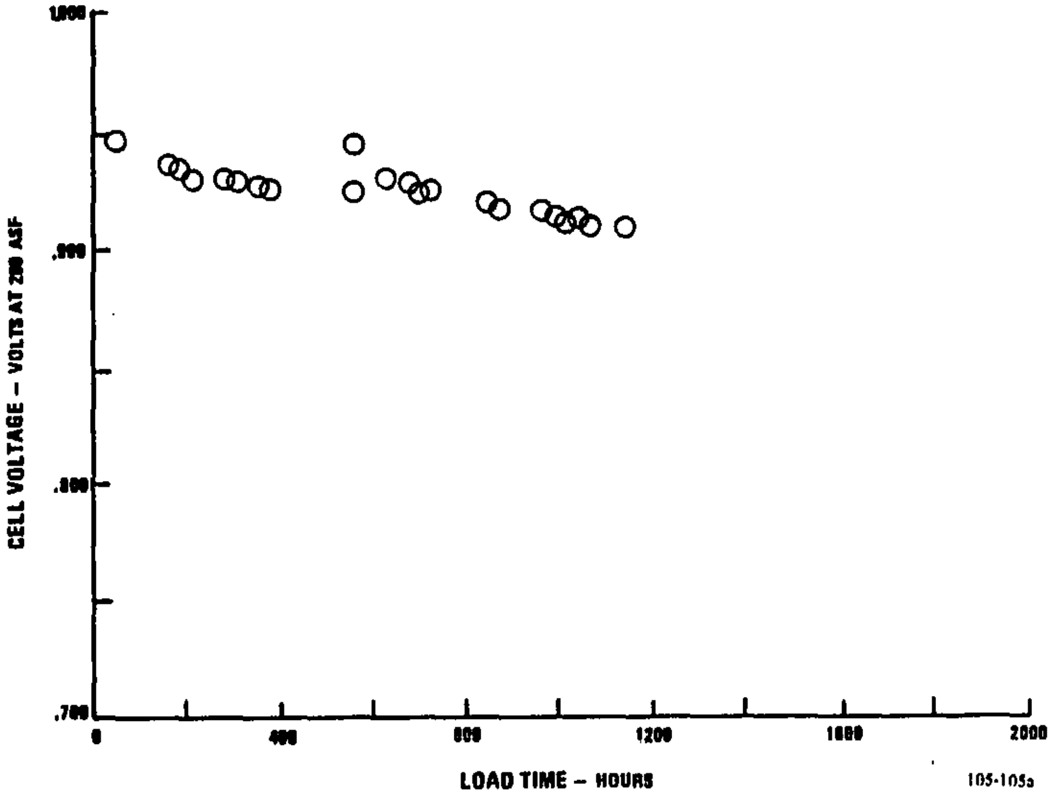


Figure 46. Research Cell RC-61 Endurance Test, 95% Potassium Titanate/5% Teflon®

Table XVI. Performance Characteristics of RC-60 and RC-61 Bonded Potassium Titanate Matrices at 250°F (121.1°C)

Research Cell	Matrix Binder	Cell Performance (Volts of 200 ASF) (215.3 mA/cm²) Initial	Cell Performance (Volts of 200 ASF) (215.3 mA/cm²) 1000 Hours	Performance Change (1000-Hrs) Cell Voltage (mV at 200 ASF) (215.3 mA/cm²)	Performance Change (1000-Hrs) Cathode Activity (mV at 10 ASF) (10.7 mA/cm²)	Performance Change (1000-Hrs) Anode Polarization (mV at 200 ASF) (215.3 mA/cm²)	Performance Change (1000-Hrs) iR (mV at 200 ASF) (215.3 mA/cm²)
RC-60	4% Butyl Rubber	0.949	0.904	45	10	27	5
RC-61	5% Teflon®	0.945	0.907	38	15	17	2

RC-62 (Bonded Potassium Titanate Matrix)

This research cell was constructed with a 95% wgt potassium titanate bonded 5% wgt Teflon® matrix. The matrix thickness was 20 mils (0.5mm), 10 mils (0.25mm) filtered onto each electrode.

The performance history of RC-62 is presented on Figure 47. The initial performance of the cell was 0.912V at 200 ASF (215.3 mA/cm²) and following 5110 hours of testing, cell voltage was 0.858V. A reduction in cell voltage at low loads had been observed in the dilute oxygen test data beginning at approximately 4000 hours of testing. This performance response was due to a slight internal electrical short or reactant cross-leakage, however, it did not preclude the successful completion of the planned 5000-hour test.

A comparison of the performance characteristics of the two long-term endurance bonded potassium titanate matrix cells is presented in Table XVII.

Table XVII. Bonded Potassium Titanate Matrix Cell Results

		Cell Voltage-V @ 200 ASF (215.3 mA/cm²)	
Cell No.	Matrix	Initial	Final
RC-54	Potassium Titanate/Butyl Rubber	0.913	0.889
RC-62	Potassium Titanate/Teflon®	0.912	0.858

RC-63 (Perforated Nickel Foil Substrate)

Research Cell RC-63 incorporated a gold-platinum catalyst layer deposited onto a gold-plated perforated nickel foil substrate. The gold-plated perforated nickel foil was evaluated under the program as a candidate lightweight, low-cost substitute for the standard 100-mesh screen. The foil being employed for this evaluation, has a

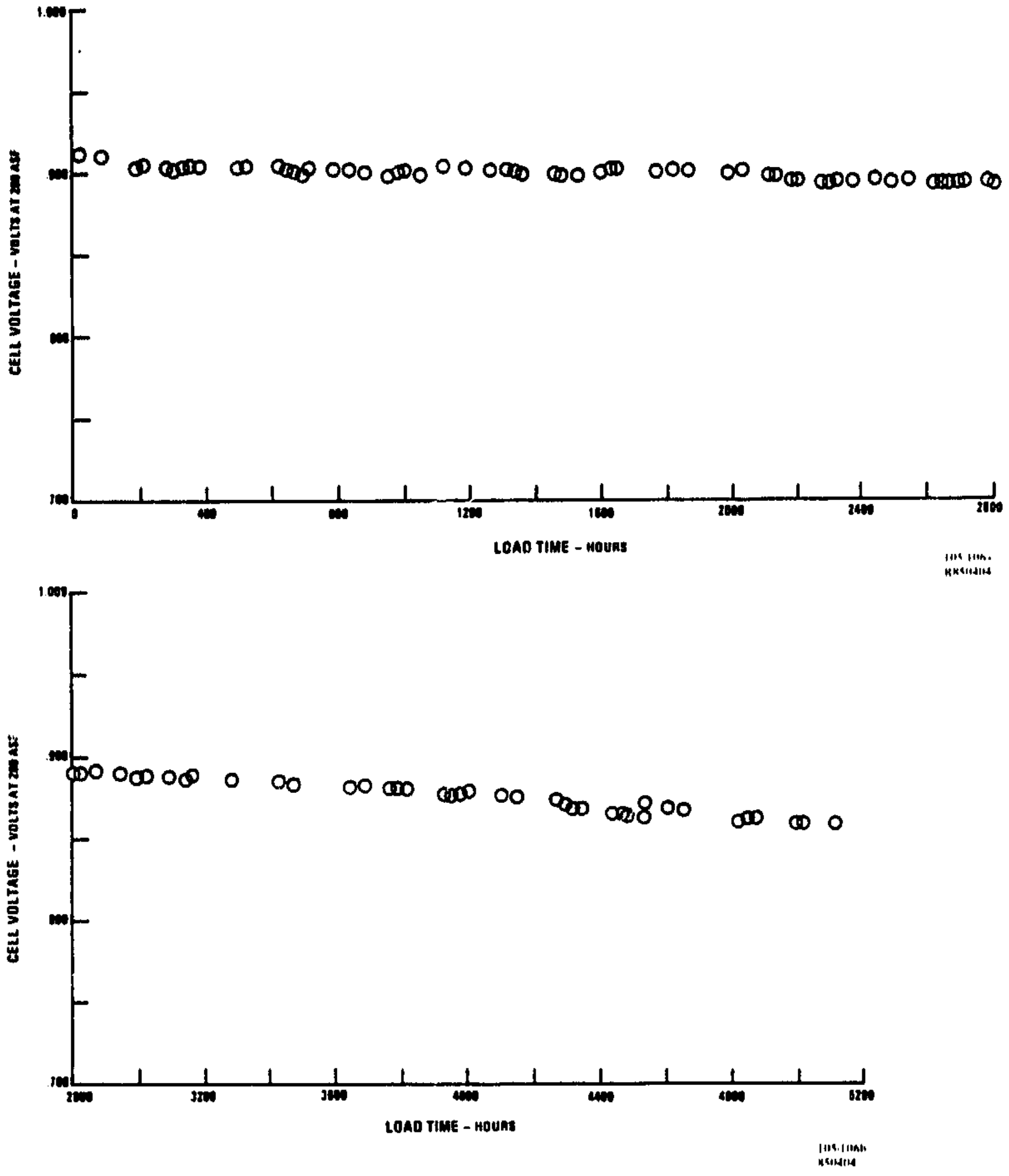


Figure 47. Research Cell RC-62 Endurance Test, Bonded Potassium Titanate Matrix

close-packed array of 25-mil diameter holes. Other details of cell construction are presented in Table XIII, Section V.

The catalyst on fine-wire mesh substrate electrodes is primarily contained in the holes between the wires, whereas, with the nickel foil, some catalyst does not penetrate the holes, but the bulk remains as a layer on the surface. As a result of this difference and the fact that the fine wire screens are wetted by electrolyte, a catalyst layer on a foil substrate may show optimum performance and endurance capability with a lower Teflon® content in the catalyst layer. Therefore RC-63 was constructed with a cathode which the catalyst Teflon® content was reduced.

RC-63 successfully completed the planned 1000-hour endurance test. A total of 1171-hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm²) and atmospheric pressure was completed. The performance history of RC-63 is presented on Figure 48.

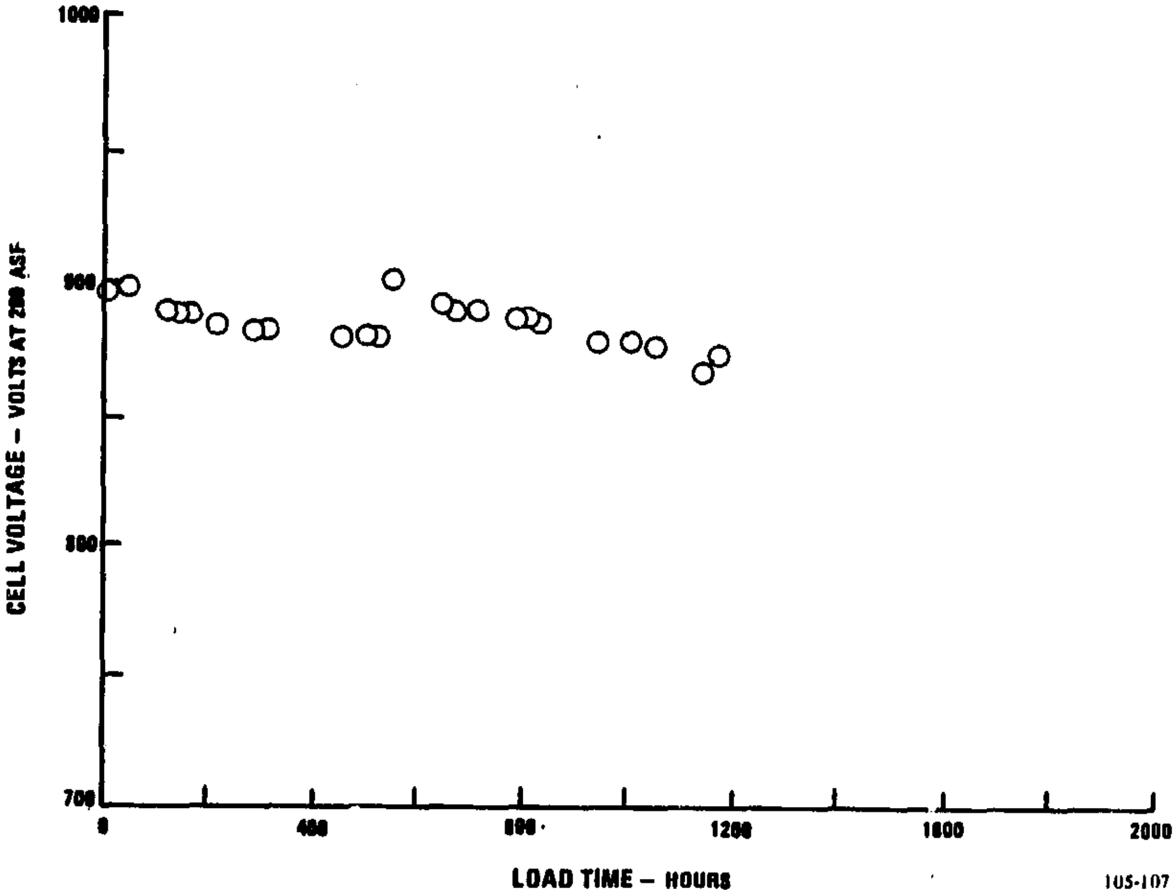


Figure 48. Research Cell RC-63 Endurance Test
(Gold-Plated Nickel Foil Substrate)

The initial performance was 0.889V @ 200 ASF (215.3 mA/cm²) and at the completion of the test, cell voltage was 0.875V. The cell voltage fall-off with time was only slightly greater than that experienced with research cells containing standard gold-platinum cathodes with fine wire mesh substrates.

RC-64 (Nafion®-Based Matrix)

Microporous Nafion® was evaluated as a candidate matrix material in a laboratory research cell. The material was in the form of a translucent sheet, 50-mils (1.3mm) thick composed of Nafion® reinforced with a Teflon® fabric. The porosity of the material was approximately 50 percent with the electrical conductivity derived primarily from the electrolyte stored within the pores.

The Nafion® matrix was filled with electrolyte by boiling the Nafion® in water for four hours and then exchanging the water with 23% wgt. KOH for 48 hours. This procedure was followed because Nafion® is very hydrophobic in the K^+ ion form. Following this electrolyte fill procedure the sheet became clear indicating that it was filled.

A 2 inch x 2 inch (5.1 cm x 5.1 cm) portion of the filled membrane was built into a subscale research cell with standard production electrodes. Other details of cell construction are presented in Table XIII, Section V.

The initial performance of the cell was very low, approximately 0.6V at 30 ASF (32.3 mA/cm²) with an iR of 180 mV at 30 ASF (32.3 mA/cm²). Varying the cell operating temperature from 170°F (76.7°C) to 180°F (82.2°C) and the oxygen inlet dewpoint temperature from 150°F (65.6°C) to 160°F (71.1°C) resulted in no significant performance improvement.

At this point in the evaluation test, RC-64 was shutdown and flush refilled with electrolyte. Following restart and upon achieving stable conditions, the maximum performance of 0.825V @ 50 ASF (53.8 mA/cm²) at a 180°F (82.2°C) cell temperature was attained. Unfortunately, this performance level was unstable and there was a corresponding increase in cell iR.

The evaluation test was interrupted for a second time. The cell was shutdown and a few gaskets were removed to increase pinch, and provide closer contact between the membrane and electrodes. The cell voltage following restart was very low. At this point the evaluation test was stopped.

A teardown inspection of RC-64 was conducted to assist in identifying the cause of the low performance. The electrodes showed some evidence of tenting which would contribute to increased cell iR. The membrane apparently lacks the resilience needed to conform to the electrodes.

RC-65 (Perforated Nickel Foil Substrate)

Research Cell RC-65 was constructed with a gold-platinum cathode catalyst layer bonded with Teflon® on a photofabricated perforated gold-plated nickel foil. The foil substrate was identical to that employed in research cell RC-63.

The performance history of RC-65 is presented on Figure 49. RC-65 successfully completed a planned 1000-hour endurance test completing 1020-hours of operation at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and one atmosphere.

In reviewing the test data, the 1000-load hour performance of RC-65 and RC-63 were identical at 0.882V at 200 ASF (215.3 mA/cm^2). Test results have shown that the perforated nickel foil is a viable substitute for the standard fine wire screen substrate. In addition, there was no significant difference in performance with catalyst layer content with the nickel foil substrates.

RC-66 (Carbon Ribbed Substrate Anode)

The carbon ribbed substrate anode is a lightweight, low-cost substitute for the fine wire electrode and nickel electrolyte reservoir plate of the alkaline fuel cell. The primary advantage however, of the configuration is the potential weight savings. Incorporating the carbon ribbed substrate into a standard production cell would result in nearly a 50 percent weight savings.

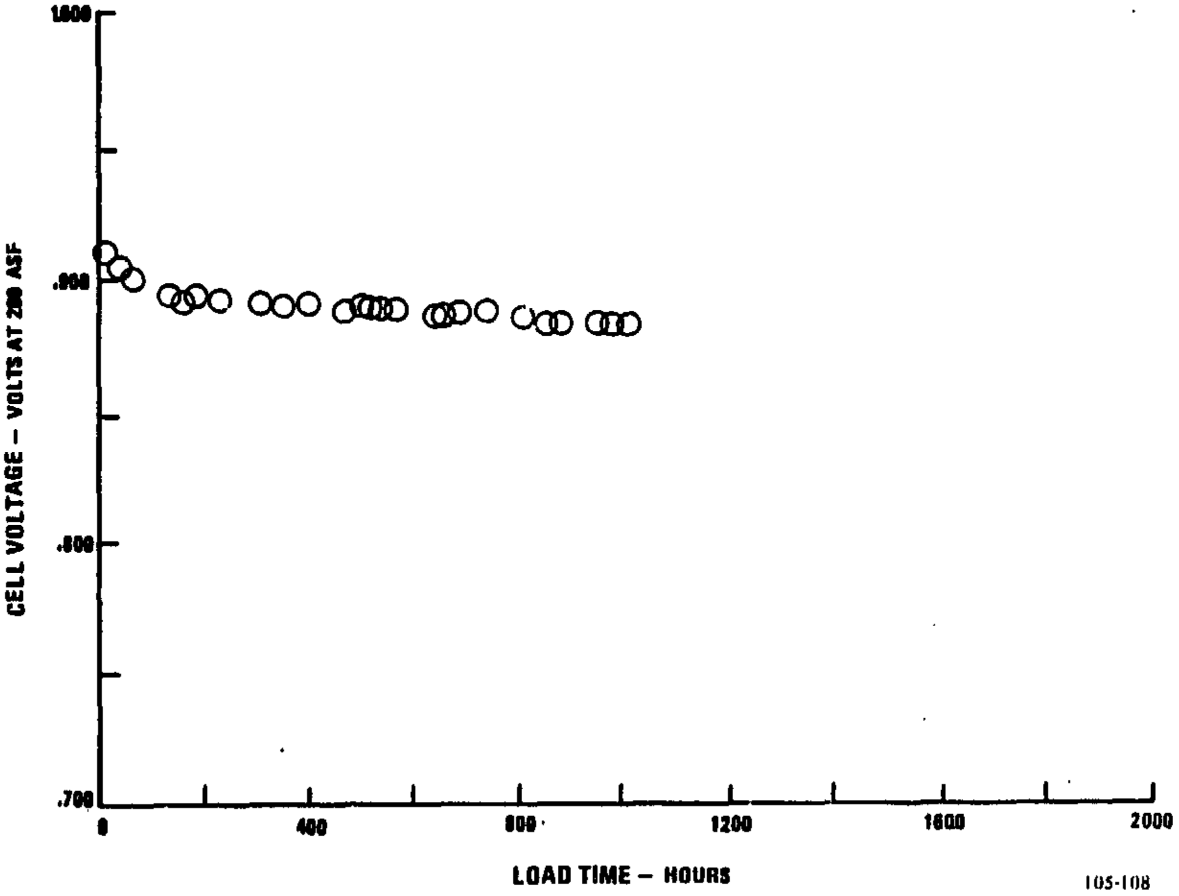


Figure 49. Research Cell RC-65 Endurance Test
Gold-Plated Nickel Foil Substrate

Research Cell RC-66 was constructed with a "dual-porosity" ribbed carbon anode substrate without hydrogen access holes. The catalyst layer consisted of platinum-palladium. Other details of cell construction is presented in Table XIII, Section V.

The performance history of RC-66 is presented on Figure 50. Over the course of the endurance test, there was little voltage fall-off. The initial cell performance was 0.899V at 200 ASF (215.3 mA/cm^2) and at completion of the test at 1010 hours the cell voltage was 0.891V at 200 ASF (215.3 mA/cm^2). The performance of RC-66 at 1010 hours when adjusted for the difference in cell iR approximately 8 MV at 200 ASF (215.3 mA/cm^2) was identical to the performance of RC-62, which contained a nickel electrolyte reservoir plate and standard fine wire screen substrate. The lower electrical conductivity of the carbon substrate results in higher cell iR.

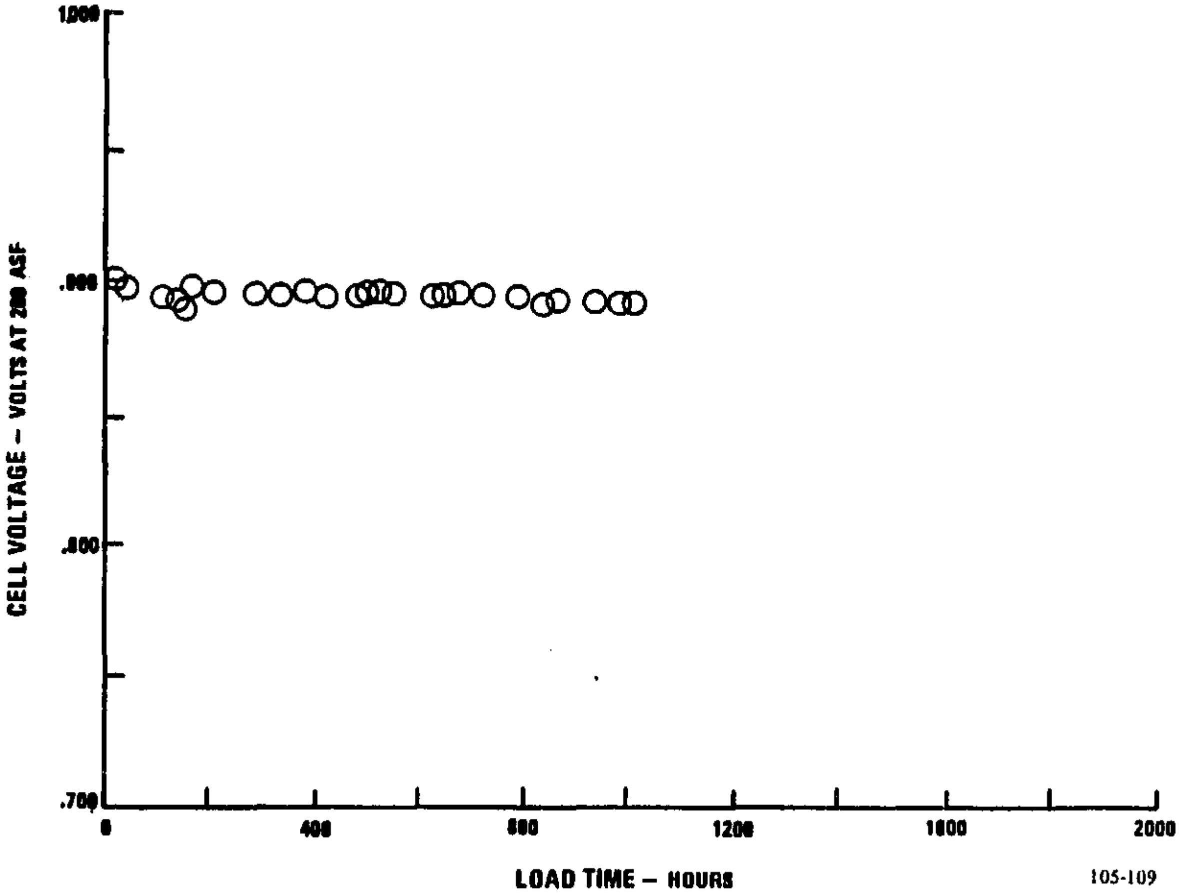


Figure 50. Research Cell RC-66 Endurance Test
(Carbon Ribbed Substrate Anode)

Research Cell RC-66 test results demonstrate the suitability of the graphite ribbed substrate configuration for use in the alkaline fuel cell.

RC-67 (Carbon Ribbed Substrate Anode)

This laboratory cell was fabricated with a "dual-porosity" carbon ribbed substrate anode and an advanced platinum-on-carbon catalyst. Details of cell construction are presented in Table XIII, Section V.

In laboratory half-cell tests, the advanced catalyst appeared easier to wet with electrolyte and does not require a hydrazine diluted electrolyte flush or any other special technique to wet the catalyst layer. Research Cell RC-67 was filled with electrolyte by the standard vacuum flush fill employed on all non-carbon substrate anode research cells.

RC-67 completed 1300-hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and atmospheric pressure. Test results were very encouraging as this was the first successful test of a carbon substrate structure which did not employ any special electrolyte fill technique.

The performance history of RC-67 is shown on Figure 51. The initial performance was 0.892V at 200 ASF (215.3 mA/cm^2) and after 1300-hours of testing the cell voltage was 0.855V at 200 ASF (215.3 mA/cm^2). There was only one performance anomaly during the test. Between 500 and 820 load hours cell voltage fell-off to 0.784V at 200 ASF (215.3 mA/cm^2). At 820 load hours the cell voltage was restored to 0.858V at 200 ASF (215.3 mA/cm^2) following an induced low cell potential, that is, nitrogen was introduced onto the cathode while at low load. This procedure may have promoted the electrolyte wetting of the anode catalyst layer.

Endurance testing of RC-67 was stopped at 1300 hours following completion of the planned 1000-hour test.

RC-68 (Carbon Ribbed Substrate)

As an approach to reducing cell iR of carbon ribbed substrate cells, Research Cell RC-68 was constructed with a thinner "web" cross-section substrate.

Initial cell iR was identical to that of RC-66, unfortunately in the subsequent 806 hours of testing cell iR gradually increased. The endurance test was stopped at 806 hours due to increasing cell iR.

Apparently the thinner web did not provide adequate structural support in the cell package.

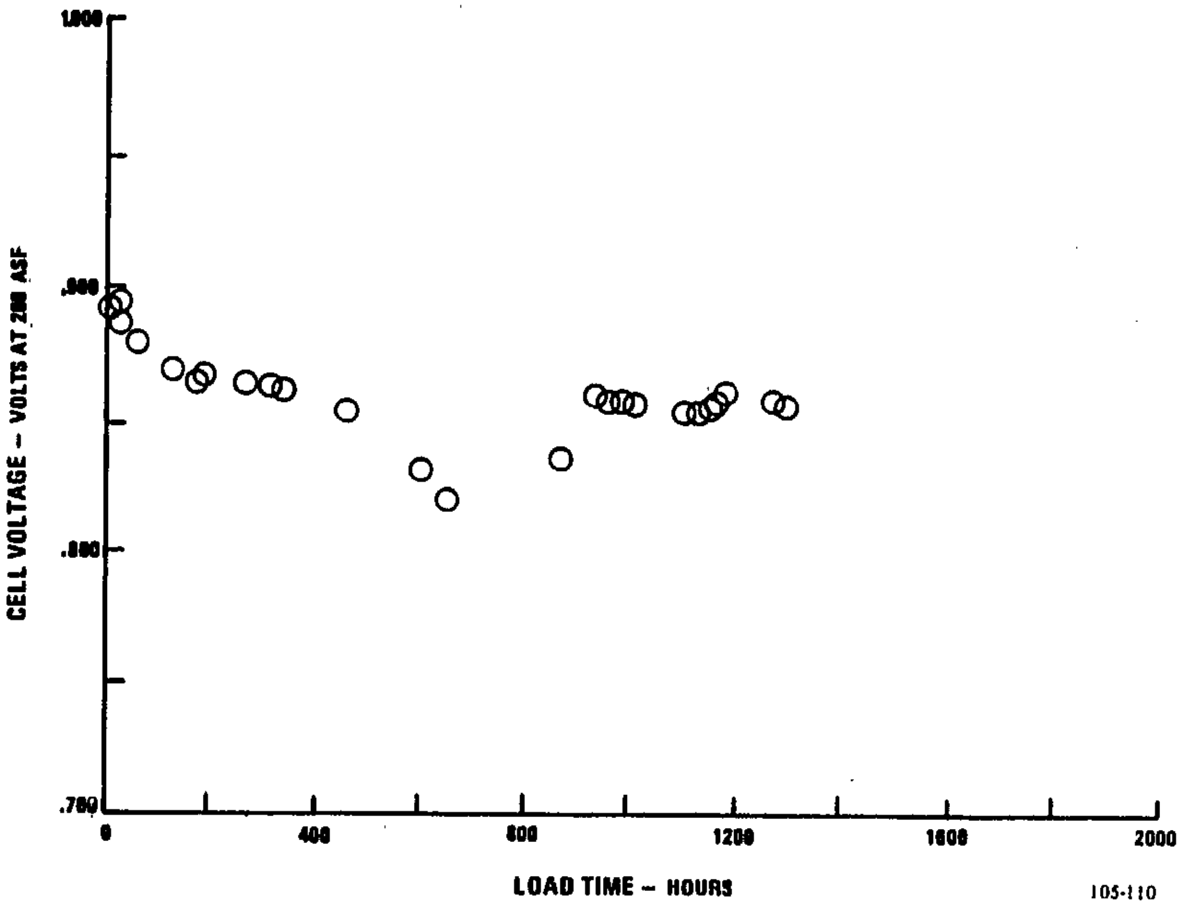


Figure 51. Research Cell RC-67 Endurance Test
(Carbon Ribbed Substrate Anode)

RC-69 (Composite Matrix)

The composite matrix consists of a potassium titanate layer deposited onto an advanced asbestos layer. The asbestos layer provides structural integrity and the high bubble pressure.

The composite matrix consists of 10-mils (0.25mm) of 95% wgt. potassium titanate 5% wgt. Teflon® deposited onto 10-mils (0.25mm) of advanced asbestos for a total matrix thickness of 20-mils (0.5mm). In RC-69 the potassium titanate layer is in contact with the anode.

RC-69 was fabricated with standard gold-platinum cathodes, platinum-palladium anodes and a 51-mil (1.3mm) thick nickel electrolyte reservoir plate.

The performance history of RC-69 is presented on Figure 52. The cell completed a total of 500-hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm²) and one atmosphere. The cell exhibited a lower than expected initial cell performance of 0.894V at 200 ASF (215.3 mA/cm²) compared to 0.912V at 200 ASF (215.3 mA/cm²) for Research Cell RC-62. In addition RC-69 exhibited a greater than expected performance and subsequent voltage fall-off led to stopping the test at 500 hours.

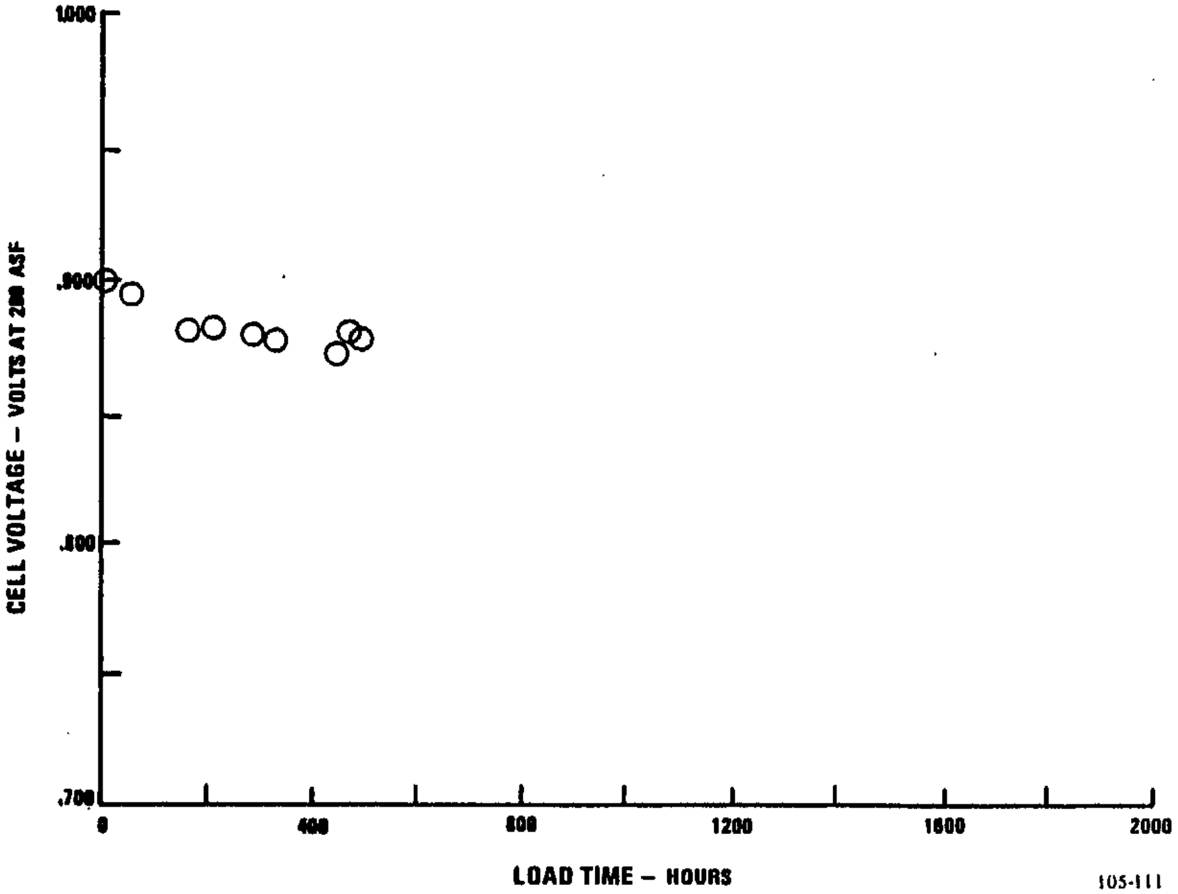


Figure 52. Research Cell (RC-69) Endurance Test Composite Matrix

RC-70 (Carbon Ribbed Substrate Anode)

Research Cell RC-70 was constructed with a "dual-porosity" nickel treated carbon ribbed substrate anode with a platinum-on-carbon catalyst layer.

Initial cell performance was lower than expected at 0.862V at 200 ASF (215.3 mA/cm^2). For comparison the initial performance of RC-67 which incorporated the same advanced platinum-on-carbon catalyst anode was 0.892V at 200 ASF (215.3 mA/cm^2).

The endurance test was continued out to 665 hour at which time the test was stopped because cell voltage was less than 0.800V at 200 ASF (215.3 mA/cm^2).

RC-71 (Composite Matrix)

RC-71 incorporated a composite matrix consisting of 10-mils (0.25mm) of 95% wgt. potassium titanate and 5% wgt. Teflon® deposited onto 10-mils (0.25mm) of advanced asbestos for a total matrix thickness of 20-mil (0.5mm). The potassium titanate layer was in contact with the anode.

The performance history of RC-71 is presented on Figure 53. The initial cell performance of 0.896V at 200 ASF (215.3 mA/cm^2) was comparable to that experienced on RC-69. The cell iR on RC-71 and RC-69 at 10 mV at 100 ASF (107.6 mA/cm^2) is consistent with past laboratory cell test experience.

RC-71 completed a planned 5000-hour test completing 5110 hours of operation at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and atmospheric pressure. At the completion of the test cell voltage was 0.782V at 200 ASF (215.3 mA/cm^2).

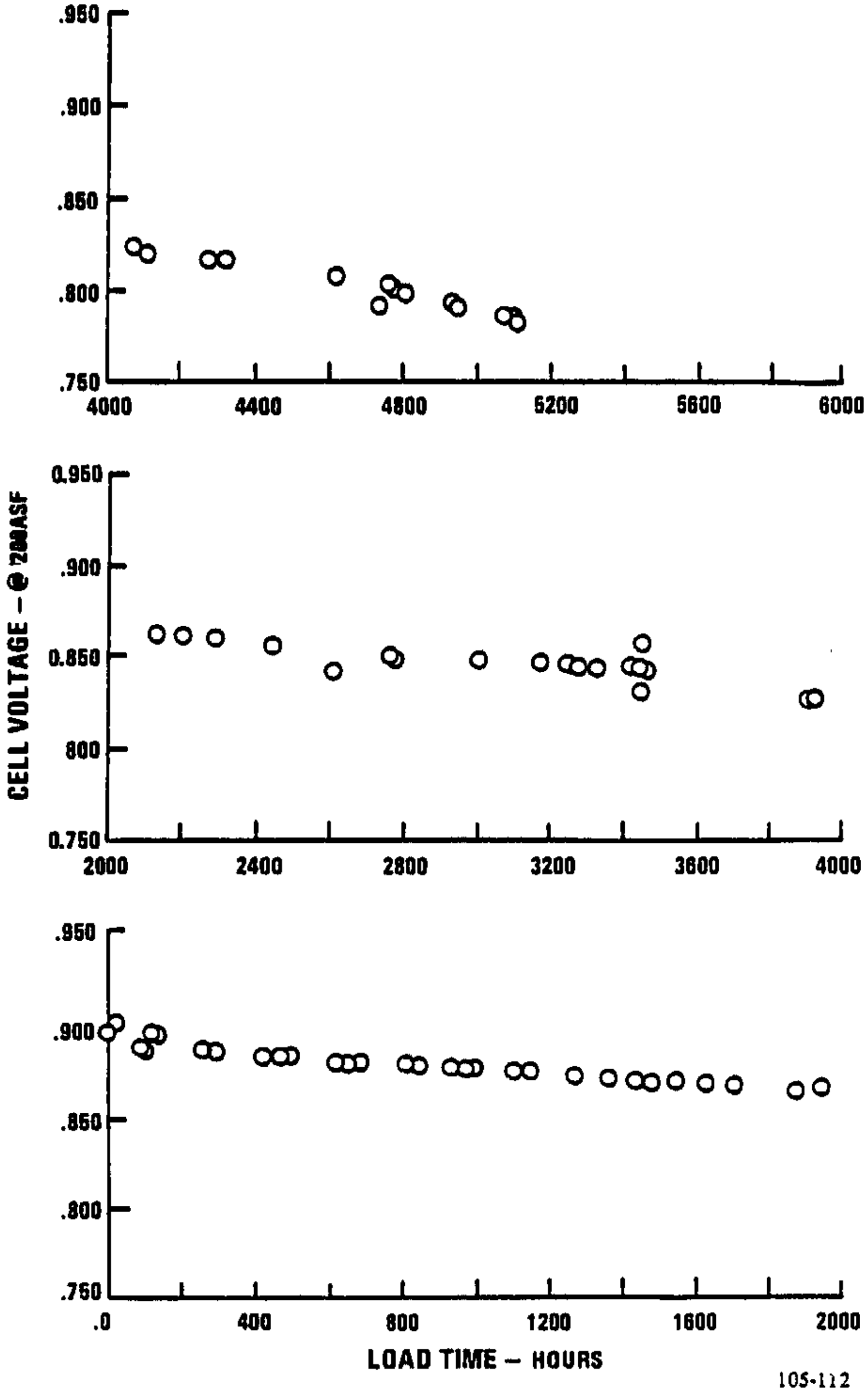


Figure 53. Research Cell (RC-71) Endurance Test Composite Matrix

A comparison of test results from Dilute Oxygen Diagnostic Tests conducted during the endurance test are summarized in Table XVIII.

Table XVIII. RC-71 Dilute Oxygen Test Results

	RC-71	RC-62*
• Cathode Activity - Volts at 10 ASF (10.8 mA/cm²)		
Initial	0.992	1.000
Final (5,000-Hours)	0.979	0.992
Change mV	13	8
• Cathode Polarization mV at 200 ASF (215.3 mA/cm²)		
Initial	5	3
Final	7	7
Change	2	4
• Anode Polarization mV at 200 ASF (215.3 mA/cm²)		
Initial	20	15
Final	100	40
Change	80	25
• Cell iR - mV at 200 ASF (215.3 mA/cm²)		
Initial	20	16
Final	32	22
Change	12	6

*Baseline Potassium Titanate Matrix Cell

The results presented in Table XVIII reveal that the majority of the performance loss experienced during the endurance test was due to an increase in anode polarization. Cathode performance was consistent with past test experience that is, there was no significant variation in activity or polarization. Cell iR increase on RC-71 was higher than normal, however, it was not a significant factor in overall cell voltage loss.

The diagnostic tests do not provide any clue to the cause of the higher than anticipated anode polarization. Two possible explanations for the increased polarization are (1) electrolyte flooding of the electrode structure with time and (2) unidentified contamination of the electrode during the test.

Upon completion of the 5,000-hour test, the cell was disassembled and visually inspected. There were no abnormalities observed in either the anode or cathode. The cathode side of the matrix appeared to have a small pit which extended about halfway through the matrix. This pit could have originated from a contaminant which dissolved over the course of the test and shifted into the anode catalyst layer and promoted increased electrode flooding with time.

RC-72 (Carbon Ribbed Substrate Anode)

RC-72 incorporated a nickel-treated carbon ribbed substrate with a platinum-on-carbon catalyst anode layer. Other details of cell construction are presented in Table XIII, Section V.

The initial cell performance of 0.831V at 200 ASF (215.3 mA/cm^2) was a disappointment. Cell iR at 13 mV at 100 ASF (107.6 mA/cm^2) although somewhat higher than expected was not a significant contributor to lower performance. Dilute oxygen diagnostic test data indicated that the majority of the lower than expected performance to be due to increased anode polarization.

The endurance test was stopped at 237 hours due to low cell performance. Cell voltage at the completion of testing was 0.797V at 200 ASF (215.3 mA/cm^2).

RC-73 (Composite Anode Structure)

The research cell was constructed with a composite anode structure comprised of a single porosity flat carbon substrate without ribs and an advanced platinum-on-carbon catalyst layer along with a standard nickel electrolyte reservoir plate.

Research Cell RC-73 completed 1300 hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and atmospheric pressure. The "best" cell performance was 0.842V and after 1300 hours cell voltage was 0.730V at 200 ASF (215.3 mA/cm^2). The test was stopped because of the inability to achieve a satisfactory performance characteristic.

RC-74 (Perforated Nickel Foil Substrate)

Research Cell RC-74 was constructed with a gold-platinum cathode catalyst layer bonded with 20% Teflon® on a photofabricated, gold plated nickel foil.

The cell successfully completed a planned 1000-hour test, accumulating 1055 hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and one atmosphere. The performance history of RC-74 is presented on Figure 54.

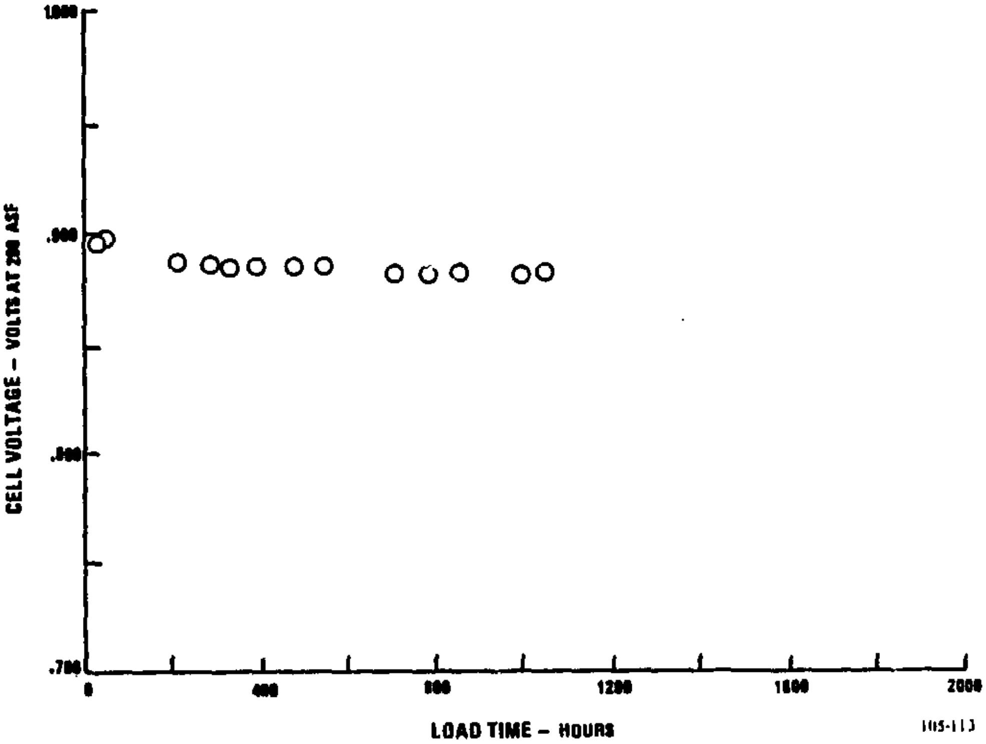


Figure 54. Research Cell (RC-74) Endurance Test (Perforated Nickel-Foil Substrate)

Table XIX presents a performance comparison of the three photofabricated nickel foil substrate cathode research cells endurance tested.

Table XIX. Photofabricated Ni-Foil Substrate Cathodes

	Catalyst Teflon® Content - %	Initial Performance V/C @ 200 ASF	Final Performance V/C @ 200 ASF	Total Time Hours	Voltage Change MV
RC-63	10	0.901	0.875	1171	26
RC-65	15	0.905	0.882	1020	23
RC-74	20	0.900	0.883	1055	17

The test data indicates that the initial performance of the three research cells was very similar. The data shows that increasing catalyst Teflon® content from 15% to 20% improves performance stability.

Based upon the laboratory research cell tests, the gold-plated, nickel photofabricated foil appears to be viable replacement for the expensive gold-plated, fine wire nickel screen.

RC-75 (Perforated Nickel Foil Substrate)

In an exploratory evaluation test, Research Cell RC-75 was constructed with a platinum-on-carbon catalyst anode with a fine wire nickel substrate. A total of 345 hours of testing was completed on RC-75.

Initial cell voltage was 0.839 V/C at 200 ASF (215.3 mA/cm^2) and following 345 hours of testing, cell performance was 0.841 V/C. Test results verify the performance stability of the platinum-on-carbon catalyst layer. The higher than expected voltage loss with time experienced on RC-73 appears to be associated with the electrode substrate.

RC-76 (Bonded CeO_2 Matrix)

The research cell was constructed with a 7-mil (0.18mm) thick butyl rubber bonded Ceria (4% wgt. butyl rubber/96% wgt. CeO_2) matrix. In addition the cell incorporated a gold-platinum catalyst cathode, a platinum-palladium catalyst anode and a 51-mil (1.3mm) porous nickel electrolyte reservoir plate.

RC-76 completed a total of 496 hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and atmospheric pressure.

Initial cell voltage was 0.900V at 200 ASF (215.3 mA/cm^2). Unfortunately a greater than expected voltage loss with time led to the test being stopped after 496 hours. Dilute oxygen diagnostic tests and a teardown inspection indicated that the cell had an internal electrical short. Visual inspection of the matrix, prior to commencement of the endurance test, identified a very dense, marginal (some cracks) matrix structure. In addition the electrolyte take-up during cell fill was lower than expected. These matrix characteristics may have contributed to the cell short.

RC-77 (Bonded Strontium Titanate Matrix)

Research Cell RC-77 was fabricated with a 6-mil (0.15mm) thick butyl rubber bonded strontium titanate (4% wgt. butyl/96% wgt. $SrTiO_3$). The cell also incorporates a gold-platinum catalyst cathode, platinum-palladium catalyst and a 51-mil (1.3mm) thick nickel electrolyte reservoir plate.

RC-77 completed 5,200-hours of testing at 200 ASF (215.3 mA/cm^2) at 200°F (93.3°C) and one atmosphere pressure.

The initial cell performance was 0.881V at 200 ASF (215.3 mA/cm^2) and after 5,200 hours of testing cell voltage was 0.825V. The performance history of RC-77 is presented on Figure 55.

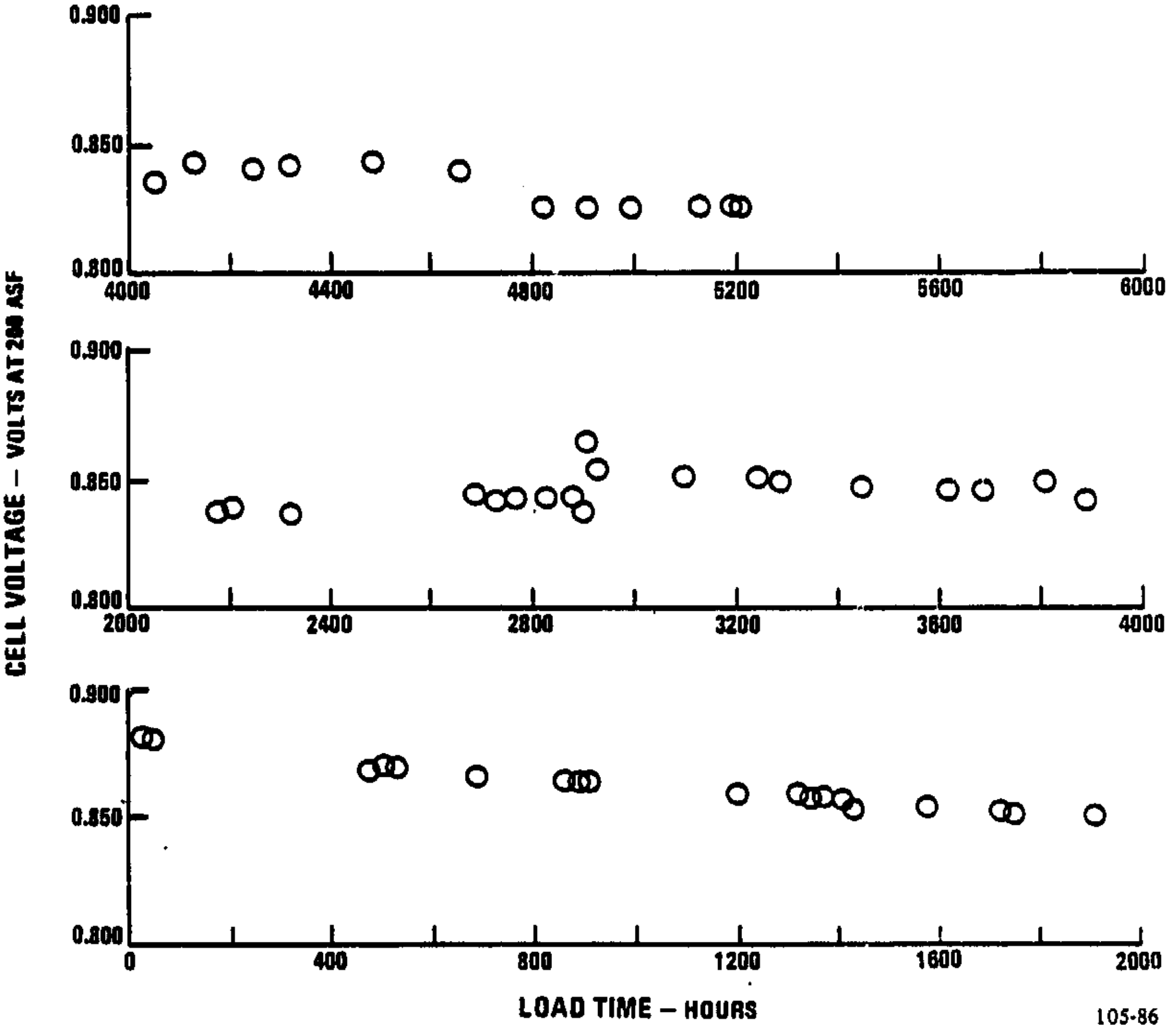


Figure 55. Research Cell (RC-77 Endurance Test
(Butyl Bonded Strontium Titanate Matrix)

Dilute oxygen test data which was collected periodically during the endurance test is summarized in Table XX.

Table XX. RC-77 Dilute Oxygen Test Data Summary

	501-Hours	5122-Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	0.988	0.986	-2 mV
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	4	8	-4 mV
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	39	83	-44 mV
Cell iR - mV @ 200 ASF	20	16	+4 mV

The test results presented in Table XX revealed that there was no significant change in cathode performance or cell iR during the endurance test. The data in Table XX indicates that the majority of the loss in cell voltage with time is due to an increase in anode polarization. One possible cause of the loss is that some unbound strontium powder may have penetrated the anode catalyst layer causing partial flooding of the electrode.

Strontium titanate, because of its chemical similarity to potassium titanate and test results from RC-77, has the potential to be an alternate matrix material. Additional matrix fabrication will be required to improve the structure. Blending the strontium titanate with potassium titanate may be an approach to improving matrix structure and bubble pressure.

RC-78 (Polybenzimidazole Matrix)

Celanese Fibers Company, Rocky Hill, SC., is producing commercial grade polybenzimidazole (PBI) fibers. PBI has been identified as a candidate matrix material on the basis of low weight loss from corrosion tests in aqueous potassium hydroxide (NASA CR-134818).

The primary advantages of PBI as a matrix material are that it contains no silicon, has a high temperature (above 250°F (121.1°C)) material stability and is fibrous, allowing fabrication of mat-type, free-standing matrices.

Evaluation of lab-made PBI samples under NASA Contract NAS3-21257 (NASA CR-159807) showed considerable variation in corrosion test results and variation in the physical makeup and color of the samples. In addition, endurance testing of research cells containing PBI matrices showed rapid voltage fall-off with time.

An exploratory test of a research cell incorporating a matrix fabricated from commercial grade PBI was initiated to re-evaluate the suitability of PBI for use in the alkaline fuel cell.

The first sample of PBI provided by Celanese was very coarse and granular. This material even when bonded with butyl rubber did not result in an acceptable matrix.

A second sample was requested from Celanese. This sample consisted of uncrimped fibers approximately 1/2 inch (1.3 cm) in length.

A matrix was fabricated by filtering a slurry of hand-cut fibers and water-alcohol mixture onto a piece of filter paper. The PBI filter cake was dried and subsequently removed from the paper forming a free standing matrix. The mat formed was 12-mils (0.30mm) thick.

The matrix was incorporated into a laboratory research cell. The cell, RC-78 was fabricated with a gold-platinum catalyst cathode, a platinum-palladium catalyst anode and a porous nickel electrolyte reservoir plate.

The performance history of RC-78 is presented on Figure 56. This cell was the first laboratory research cell with a PBI matrix to operate for 1000-hours. The best performance attained was 0.896 volts at 100 ASF (107.6 mA/cm^2). Due to voltage loss, the cell was not capable of maintaining a 100 ASF (106.6 mA/cm^2) current density and after 1205-hours of testing, cell voltage was 0.827V at 30 ASF (31.8 mA/cm^2). The inadequate performance characteristics which precluded the obtaining useful diagnostic test data was suspected to be caused by contamination of the catalyst(s) by some decomposition product from the organic fiber.

Test results were consistent with previous research cell tests of PBI matrices and the material is unsuitable for use as a matrix in the alkaline fuel cell.

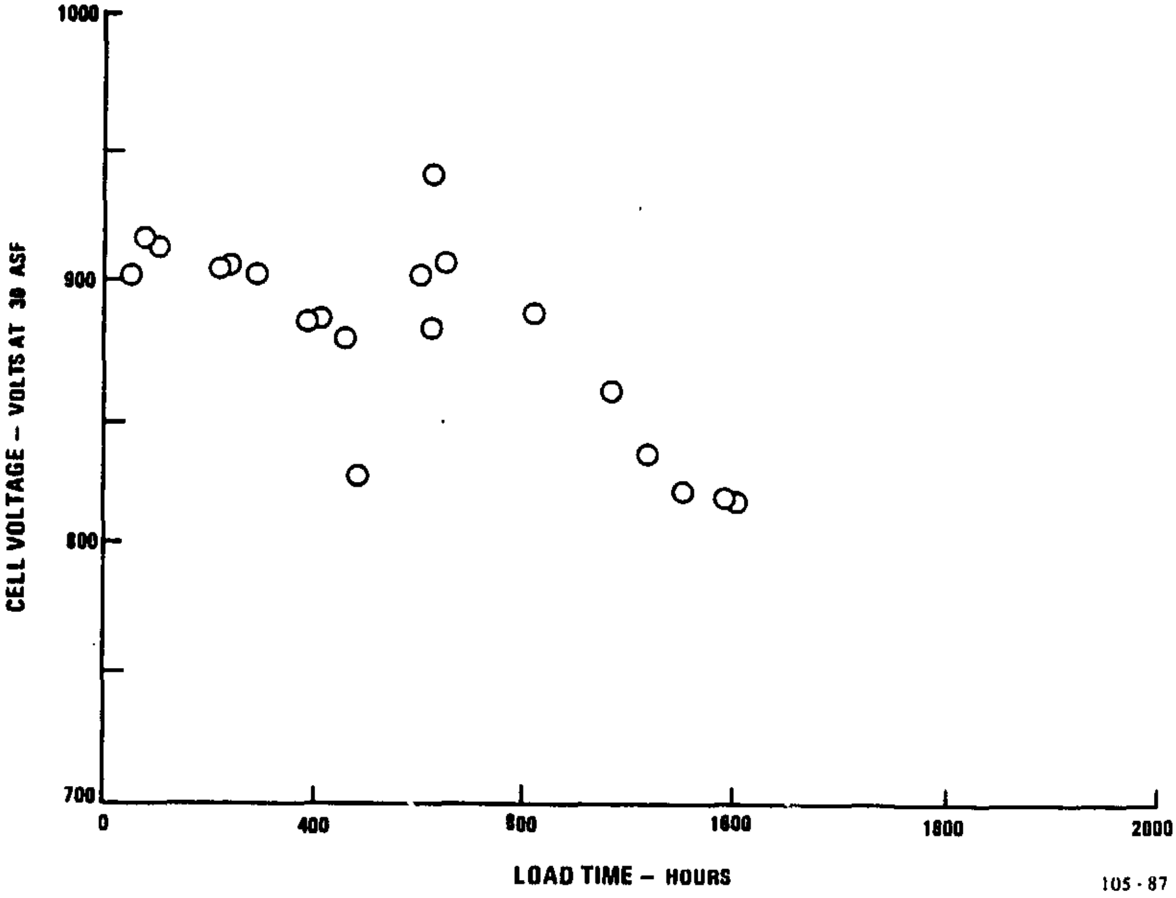


Figure 56. Research Cell (RC-78 Endurance Test
(Lab Fabricated PBI Matrix)

RC-79 (Carbon Ribbed Substrate Anode)

Research Cell RC-79 was the first cell in a series of planned cell tests to provide data to help identify a carbon substrate structure with the most desirable characteristics.

RC-79 was constructed with a gold-platinum catalyst cathode, a 20-mil (0.5mm) thick advanced asbestos matrix and a platinum-palladium anode catalyst layer deposited onto a carbon ribbed substrate.

The performance history of the cell is presented on Figure 57.

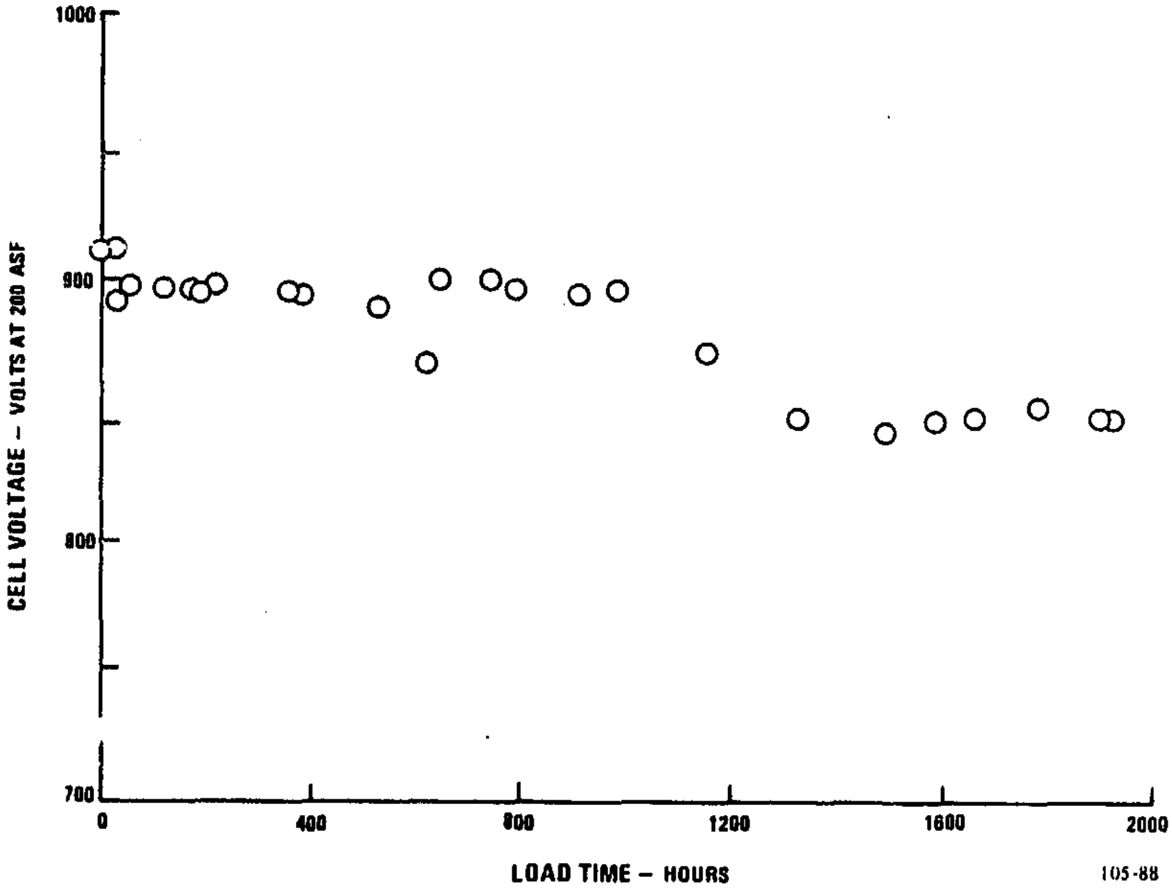


Figure 57. Research Cell (RC-79) Endurance Test (Carbon Substrate Anode)

Research Cell RC-79 was rebuilt at 620 hours with new electrodes because examination of the cell at 620 hours revealed that it was excessively dry and the cause was traced to a faulty test stand fuel flow valve. Intermittent high gas flow rates had caused the electrolyte to dry out. The valve was replaced and the cell was rebuilt with new electrodes.

The initial cell performance after rebuild was 0.899V at 200°F (93.3°C) and 200 ASF (215.3 mA/cm²). The final performance after 1300-hours of testing was 0.846V at 200 ASF (215.3 mA/cm²).

Table XXI summarizes test data from the dilute oxygen diagnostic conducted during the endurance test.

Table XXI. RC-79 Dilute Oxygen Test Data Summary

	24 Hours	1176 Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	1.009	0.992	-17
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	8	5	+3
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	19	43	-24
Cell iR - mV @ 200 ASF (215.3 mA/cm²)	22	30	-8

The data presented in Table XXI reveals that the majority of the voltage loss during the endurance test was due to a reduction in cathode activity and an increase in anode polarization.

RC-80 (Carbon Ribbed Substrate Anode)

RC-80 incorporated a carbon ribbed anode substrate with a catalyst layer of platinum-palladium. The matrix was advanced asbestos and the cathode was a standard production gold-platinum catalyst layer on a gold-plated, fine-wire nickel screen.

The cell forms part of a series of tests to evaluate substrates having different porosities and pore spectra to identify a carbon substrate which has the most desirable characteristics in regard to conductivity, diffusivity and electrolyte transfer capability. In RC-80 the rib and web densities are similar.

To improve substrate wettability, the substrates were given a nickel treatment before applying the catalyst layer.

The performance history of RC-80 is presented in Figure 58. The initial cell performance was 0.906V at 200 ASF (215.3 mA/cm²) and 200°F (93.3°C). The cell successfully completed a planned 1000-hour test accumulating 1200-hours of testing. The cell voltage at shutdown was 0.885V at 200 ASF (215.3 mA/cm²) and 200°F (93.3°C).

Dilute oxygen test data collected periodically during the endurance test is summarized in Table XXII.

The goal of the test series, based upon dilute oxygen test data and endurance test results will be to identify an improved carbon substrate structure.

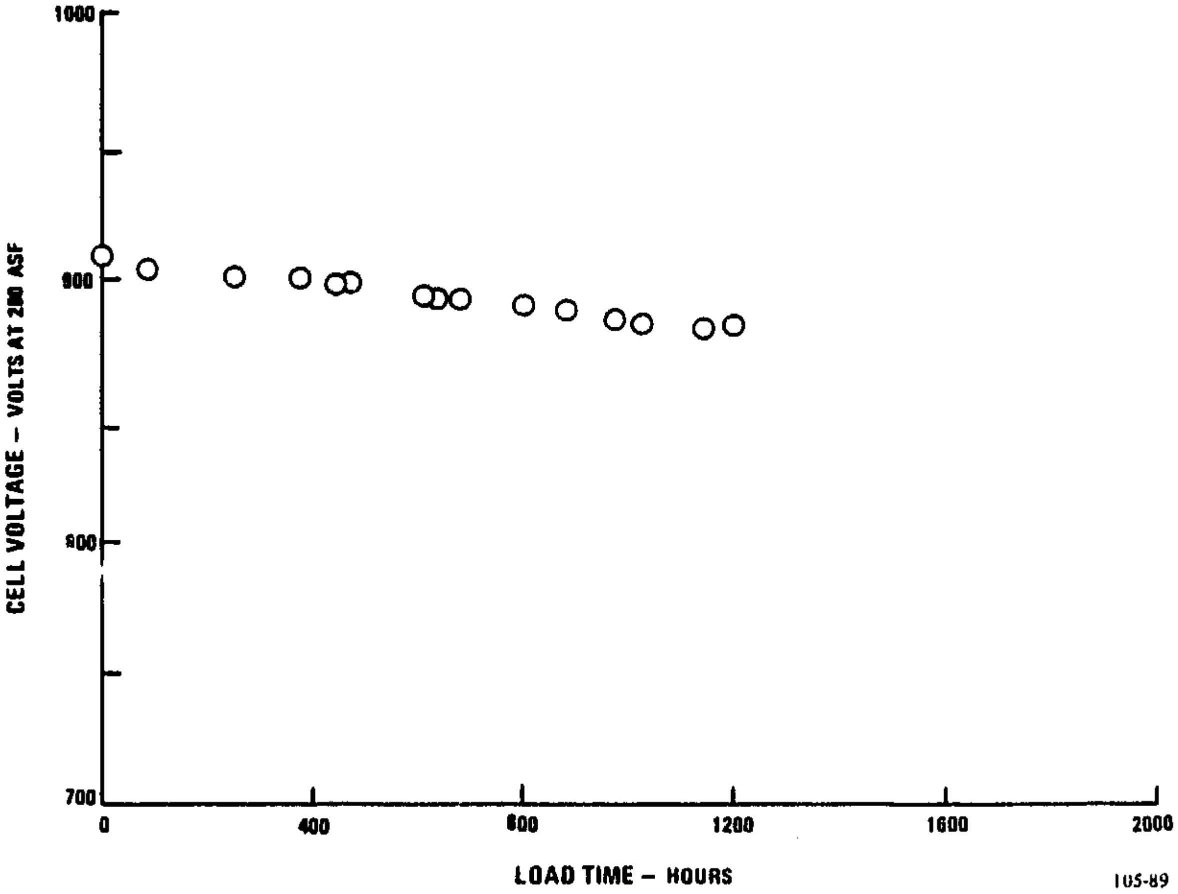


Figure 58. Research Cell (RC-80) Endurance Test

Table XXII. RC-80 Dilute Oxygen Test Data Summary

	377 Hours	1295 Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	1.003	0.995	-8 mV
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	2	7	-5 mV
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	24	43	-19 mV
Cell iR - mV @ 200 ASF (215.3 mA/cm²)	24	26	-2 mV

RC-81 (Bonded Polyantimonic Acid Matrix)

Research Cell RC-81 was the first cell to incorporate a bonded polyantimonic acid matrix. RC-83 was fabricated with a standard gold-platinum catalyst cathode, a platinum-palladium catalyst anode and a nickel electrolyte reservoir plate.

A sample of polyantimonic acid (PAA) was prepared in the laboratory from a procedure outlined in the literature. The material produced was similar in appearance to potassium titanate powder. NASA-Lewis had suggested that PAA might be a candidate matrix material based upon its reported chemical stability in hot aqueous potassium hydroxide.

In this exploratory test, the matrix was formed by depositing a layer of 95% PAA/5% Teflon® onto both electrodes. The electrodes with the still "wet" matrix were assembled into a cell and flush filled with electrolyte.

The cell was mounted on the test bench for a short performance evaluation test. The initial open circuit voltage was low and within an hour the test was stopped. Reactant cross pressure tests indicated that the cell had developed internal reactant cross leakage.

To improve the PAA matrix structure and help improve bubble pressure, the next cell (RC-82) was constructed with a PAA matrix employing asbestos as a binder.

RC-82 (Bonded Polyantimonic Acid Matrix)

This cell incorporated a free-standing fiber reinforced polyantimonic acid (PAA) matrix. The reinforcing fiber was asbestos. The cell incorporated a standard gold-platinum catalyst cathode, a standard platinum-palladium catalyst anode and a porous nickel electrolyte reservoir plate.

The initial cell performance was very low at 0.850V at 100 ASF (107.6 mA/cm^2). In the subsequent 6-hours of testing cell voltage had fallen to less than 0.5V. At this point the evaluation test was stopped. There was no evidence from the test that the cell was experiencing reactant cross leakage or internal cell shorting.

There are two possible explanations for the loss in cell voltage, (1) a loss of cell structural integrity leading to an increase in cell iR and (2) the possible contamination of either or both electrodes by decomposition products of the polyantimonic acid.

No further research cell tests of PAA matrices were conducted under the program. An acceptable matrix structure with adequate bubble pressure and chemical stability will have to be fabricated before the PAA matrix can be evaluated in laboratory research cells.

RC-83 (Bonded Potassium Titanate Matrix)

Research cell RC-83 was constructed to assist in identifying an electrolyte fill procedure on cells to be incorporated into the NASA-Lewis Four-Cell Stack 5000 Hour Test Rig 39673-1. The test summary of the stack test is presented in Section III.

RC-83 was constructed with a platinum-on-carbon catalyst anode, a butyl-bonded, potassium titanate matrix, a gold-platinum catalyst cathode and a 51-mil thick porous nickel electrolyte reservoir plate. The matrix in RC-83 was from the fabrication lot made for the four-cell stack.

A preliminary fill plan was defined based upon test results from half-cell performance tests of 1 cm x 1 cm area cathode and anode samples. Electrode samples were immersed in dilute solutions of isopropanol and water. Following this prewetting setup, the samples were filled with aqueous potassium hydroxide and half-cell tested. Test results indicated that a prewet solution of 15% isopropanol wetted the structure sufficiently without promoting electrolyte flooding of the electrode.

RC-83 was filled with a 15% isopropanol/water solution and allowed to soak for one hour. Following this step, the cell was flush filled with 23% wgt. KOH. A bubble pressure measurement of the cell, showed a limit of 13 psi (9.0 N/cm^2).

The initial performance of the cell was low at 0.799V at 200 ASF (215.3 mA/cm^2). At open circuit, a slight consumption of oxygen was observed on the flowmeter, which is an indication of an electrical short. The test was stopped after 184-hours because an acceptable performance level could not be attained. The final cell voltage was 0.821V at 200 ASF (215.3 mA/cm^2).

A bubble pressure measurement of the cell following completion of the test showed a limit of 12 psi (8.3 N/cm^2).

The isopropanol pre-wetting approach to achieve an adequate fill appears to be unsuitable. The next cell (RC-84) was filled with electrolyte utilizing the laboratory hydrazine procedure employed on cells containing platinum-on-carbon catalyst anodes.

RC-84 (Bonded Potassium Titanate Matrix)

RC-84 was the second laboratory cell employed to assist in defining an electrolyte fill procedure for four-cell stacks. Cell construction was identical to RC-83.

The cell was filled with electrolyte by following the laboratory hydrazine diluted electrolyte fill procedure. The first step in the fill procedure is to flush the cell with hydrazine diluted electrolyte. Following completion of the electrolyte flush, the unit is subjected to a thermal cycle to operating temperature. The last step in the fill procedure is to replace the hydrazine diluted electrolyte with 23% wgt. KOH.

The bubble pressure of the filled cell exceeded 21 psi (14.5 N/cm^2).

The initial performance was good at 0.892V at 200 ASF (215.3 mA/cm^2). Following 50-hours of testing, cell voltage had remained unchanged at 0.892V. Testing was stopped because the laboratory fill procedure was suitable for use on the four-cell stack.

The bubble pressure of the cell at the completion of the test was 38 psi (26.2 N/cm^2). The high bubble pressure verifies that the electrolyte had thoroughly wetted the matrix, thereby providing a very adequate barrier to prevent reactant cross mixing.

RC-85 (Carbon Ribbed Substrate Anode)

The cell was constructed with a standard gold-platinum catalyst cathode, a 20-mil (.5mm) thick advanced asbestos matrix, a platinum-palladium catalyst anode deposited onto a dual porosity carbon ribbed substrate.

The initial cell performance was 0.882V at 200 ASF (215.3 mA/cm^2) and upon completion of the test cell voltage was 0.849V at 200 ASF (215.3 mA/cm^2). The performance history of RC-85 is presented on Figure 59.

Dilute oxygen test data is summarized in Table XXIII.

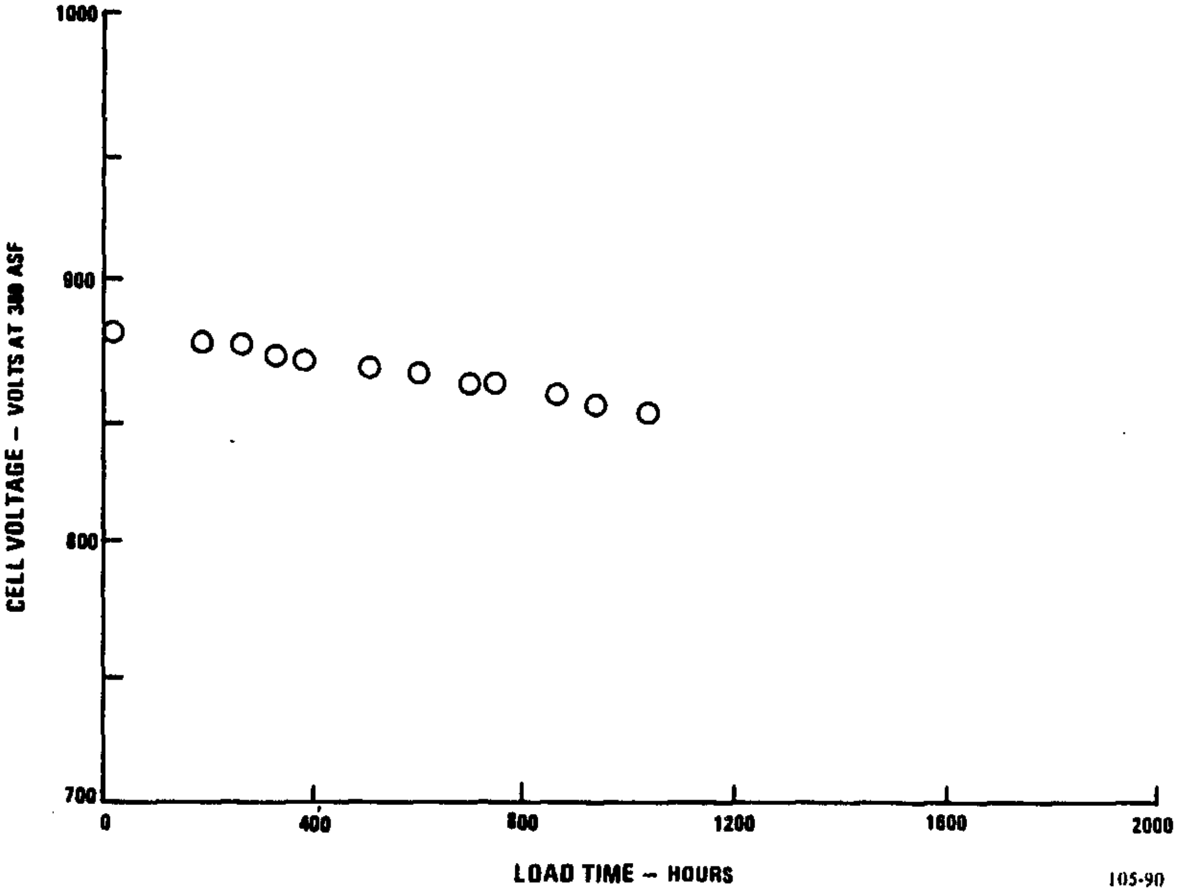


Figure 59. Research Cell (RC-85) Endurance Test
(Carbon Substrate Anode)

Table XXIII. RC-85 Dilute Oxygen Test Data Summary

	166-Hours	1035-Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	1.003	.947	56
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	2	5	3
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	39	48	9
Cell iR - mV @ 200 ASF (215.3 mA/cm²)	24	26	2

The reduction in cathode activity was responsible for the voltage loss during the endurance test. Teardown inspection of the cell did not identify any cause for the loss of activity.

RC-86 (Carbon Ribbed Substrate Anode)

Two varieties of dual porosity substrate, designated as 6W and 20W were evaluated under this program. In the 6W material the ribs are about twice as dense as the webs whereas in the 20W material the ribs and webs have similar densities. Both types of substrates were treated with nickel to render them wettable prior to applying the catalyst layers.

The anode catalyst is 10% Pt supported on carbon at a loading of 0.5 mg Pt/cm² electrode. The percentage of Teflon® in the anode catalyst layers and also the sintering conditions are being varied to determine what effect these have on performance and voltage loss with time.

RC-86 was constructed with a 10% Pt supported on carbon catalyst anode layer on a nickel treated dual porosity carbon-ribbed substrate (20W). The cathode was a standard production gold-platinum catalyst electrode. The matrix was 20-mil (.5mm) thick advanced asbestos.

The performance history of RC-86 is presented on Figure 60. The initial cell performance was 0.878V at 200 ASF (215.3 mA/cm²) and at the completion of the endurance test at 1155-hours cell voltage was 0.861V.

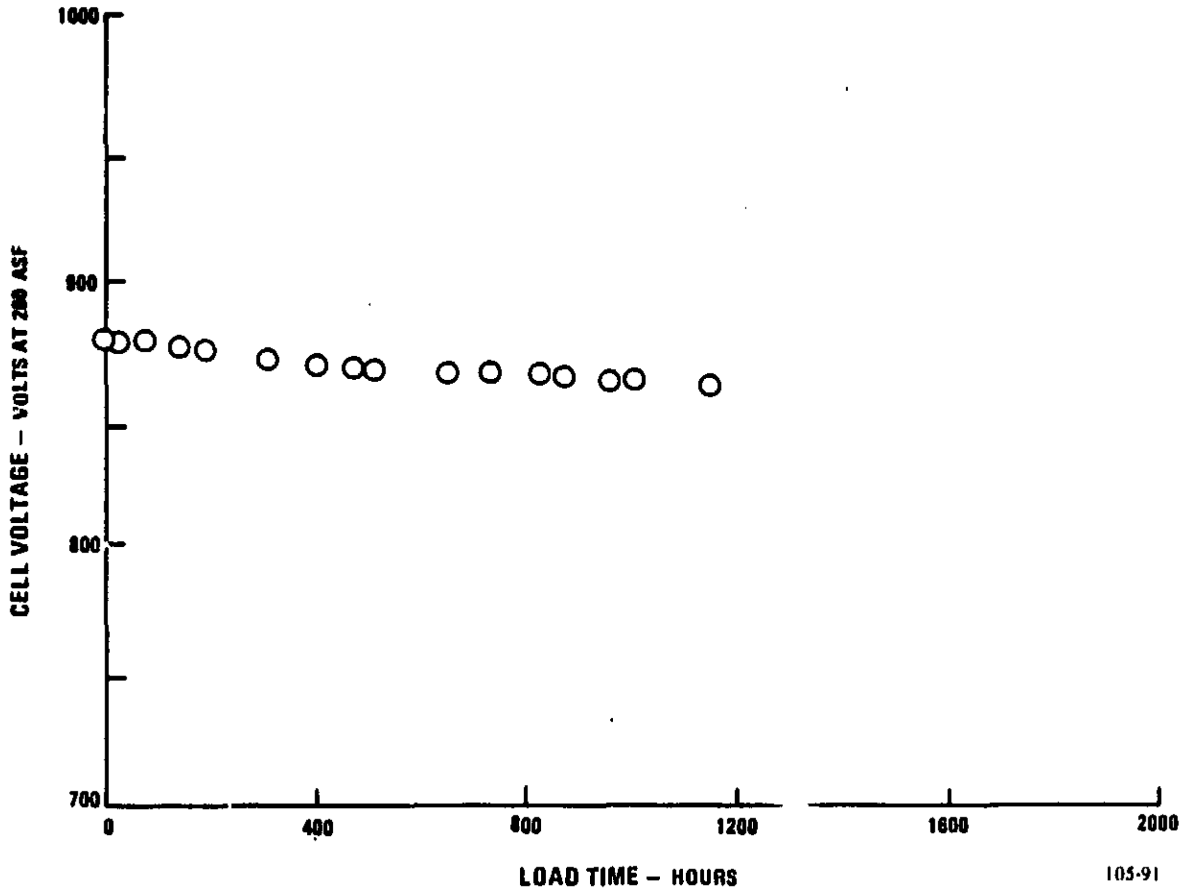


Figure 60. Research Cell (RC-86) Endurance Test (Carbon-Ribbed Substrate Anode)

A summary of dilute oxygen test data obtained during the test is summarized in Table XXIV.

Table XXIV. RC-86 Dilute Oxygen Test Data Summary

	Initial (29 Hours)	Final (1030 Hours)	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	.996	.994	- 2mV
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	5	2	+ 3mV
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	31	44	-13mV
Cell iR - mV @ 200 ASF (215.3 mA/cm²)	26	28	- 2mV

Test results presented in Table XXIV indicate that the majority of the cell voltage loss with time was due to anode polarization increase.

RC-87 (Carbon Ribbed Substrate Anode)

RC-87 was built as a replacement to RC-85. The cell incorporated a ribbed substrate anode in which the web and rib had a wide difference in density.

The cell completed a planned 500-hour endurance test. The initial cell voltage was 0.872V at 200 ASF (215.3 mA/cm²) and at the completion of the test (500-hours), cell performance was 0.844V.

A summary of dilute oxygen test data obtained during the test is presented in Table XXV.

Table XXV. RC-87 Dilute Oxygen Test Data Summary

	44-Hours	483-Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm²)	1.004	.997	- 7
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm²)	3	3	0
Anode Polarization - mV @ 200 ASF (215.3 mA/cm²)	43	69	- 26
Cell iR - mV @ 200 ASF (215.3 mA/cm²)	28	28	0

As expected the majority of cell voltage loss was due to anode polarization increase.

RC-88 (Carbon Ribbed Substrate Anode)

The laboratory cell was constructed with a carbon ribbed anode substrate in which the rib and web had similar densities. The cell incorporated a standard gold-platinum catalyst cathode, a 20-mil (.5mm) thick advanced asbestos matrix and a platinum-on-carbon catalyst anode.

RC-88 completed a planned 500-hour test completing 505-hours of testing at 200°F (93.3°C), 200 ASF (215.3 mA/cm^2) and atmospheric pressure. Initial cell voltage was 0.870V at 200 ASF (215.3 mA/cm^2) and at completion of testing cell voltage was 0.853V.

Table XXVI presents a summary of dilute oxygen test data obtained during the endurance test.

Table XXVI. RC-88 Dilute Oxygen Test Data Summary

	192-Hours	390-Hours	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm^2)	.995	.997	+ 2
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm^2)	7	7	0
Anode Polarization - mV @ 200 ASF (215.3 mA/cm^2)	48	50	- 2
Cell iR - mV @ 200 ASF (215.3 mA/cm^2)	26	26	0

Of all the laboratory screening tests of carbon ribbed substrates, RC-88 has demonstrated the smallest increase in anode polarization.

RC-89 (Compound Electrode Structure)

An exploratory test of a new compound catalyst anode structure was undertaken to evaluate the potential of the structure to extend the operating life of the alkaline fuel cell.

The structure shown in Figure 61, consists of a porous carbon layer deposited on to the matrix side of the platinum catalyst anode.

The carbon layer of the compound electrode structure is intended to perform the function of an electrolyte purifier. Contaminants in the electrolyte resulting from the chemical and physical breakdown of the plastic cell edge frame and the electrochemical decomposition of the matrix, are to be removed in the carbon layer as the electrolyte transfers between the cell and the electrolyte reservoir plate. By

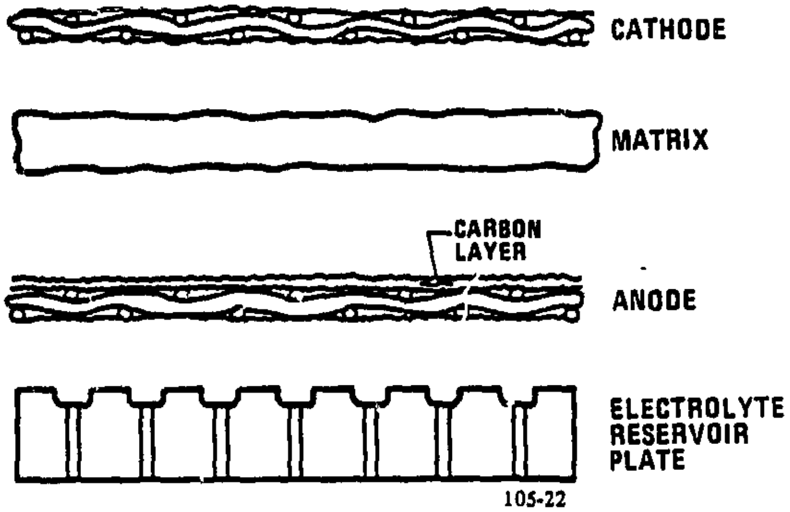


Figure 61. Compound Electrode Structure

removing these contaminants, which are suspected of promoting increased anode polarization losses, cell operating life would be extended.

To evaluate this concept, RC-89 was constructed with a standard gold-platinum catalyst anode, 20-mil (.5mm) thick advanced asbestos matrix, a standard platinum-palladium catalyst anode with a 3-mil (.08mm) thick carbon "getter" layer adjacent to the matrix, and a 51-mil (1.3mm) thick nickel electrolyte reservoir plate.

The performance history of RC-89 is presented on Figure 62. The initial cell voltage was 0.889V at 200 ASF (215.3 mA/cm^2) and following 1159-hours of testing the cell performance is 0.879V.

Cell performance was reasonably stable, however the performance level was lower than expected. Anode polarization losses were approximately 7mV higher than expected and are approximately 14mV higher than expected after 1159-hours of testing. The test goal is to complete 2000-hours of operation.

A summary of dilute oxygen test data collected during the test is presented in Table XXVII.

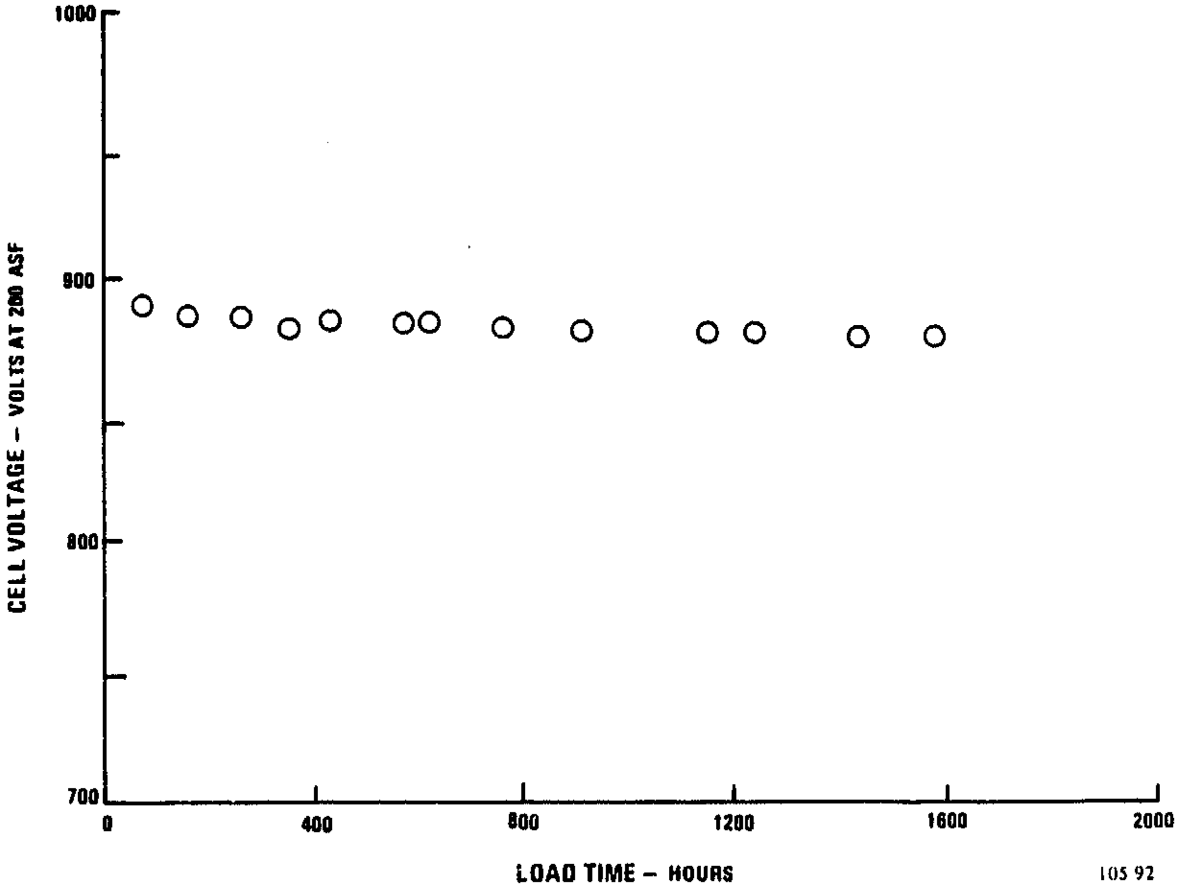


Figure 62. Research Cell (RC-89) Endurance Test (Compound Platinum Catalyst Anode)

Table XXVII. RC-89 Dilute Oxygen Test Data Summary

	Initial (77-Hours)	Present (1159-Hours)	Change
Cathode Activity - V @ 10 ASF (10.7 mA/cm^2)	1.005	1.000	- 5mV
Cathode Polarization - mV @ 200 ASF (215.3 mA/cm^2)	7	4	+ 3mV
Anode Polarization - mV @ 200 ASF (215.3 mA/cm^2)	23	37	-14mV
Cell iR - mV @ 200 ASF (215.3 mA/cm^2)	22	18	+ 4mV

The endurance test continues.

RC-90 (Carbon Ribbed Substrate Anode)

This research cell was constructed with a 10% Pt supported on carbon anode catalyst layer on a nickel treated dual porosity carbon-ribbed substrate (20W). The anode catalyst layer contained more Teflon® than the catalyst layer in RC-86. The cell contained a standard gold-platinum catalyst cathode. The matrix was 20-mil (.5mm) thick advanced asbestos.

The performance history of RC-90 is presented on Figure 63. The initial cell voltage was 0.876V at 200 ASF (215.3 mA/cm²) and after 982-hours of testing cell performance was 0.865V at 200 ASF (215.3 mA/cm²).

Initial dilute oxygen diagnostic tests indicated that the anode polarization for the cell was higher than expected. However, this has improved with time and most of the cell's voltage loss during this period is attributable to a reduction in cathode activity. The cell will continue on endurance as long as it appears stable or until 2,000 hours is reached.

RC-91 (Carbon Ribbed Substrate Anode)

RC-91 was constructed with a 10% Pt/C anode catalyst layer deposited on a nickel treated dual porosity carbon ribbed substrate (6W). The cell incorporates a standard gold-platinum catalyst cathode. The cell matrix is 20-mil (.5mm) thick advanced asbestos.

The initial performance was 0.846V at 200 ASF (215.3 mA/cm²). The inability to achieve an acceptable performance level led to stopping the endurance test after 292-hours of operation.

Research cell RC-93 was fabricated as a replacement to this cell.

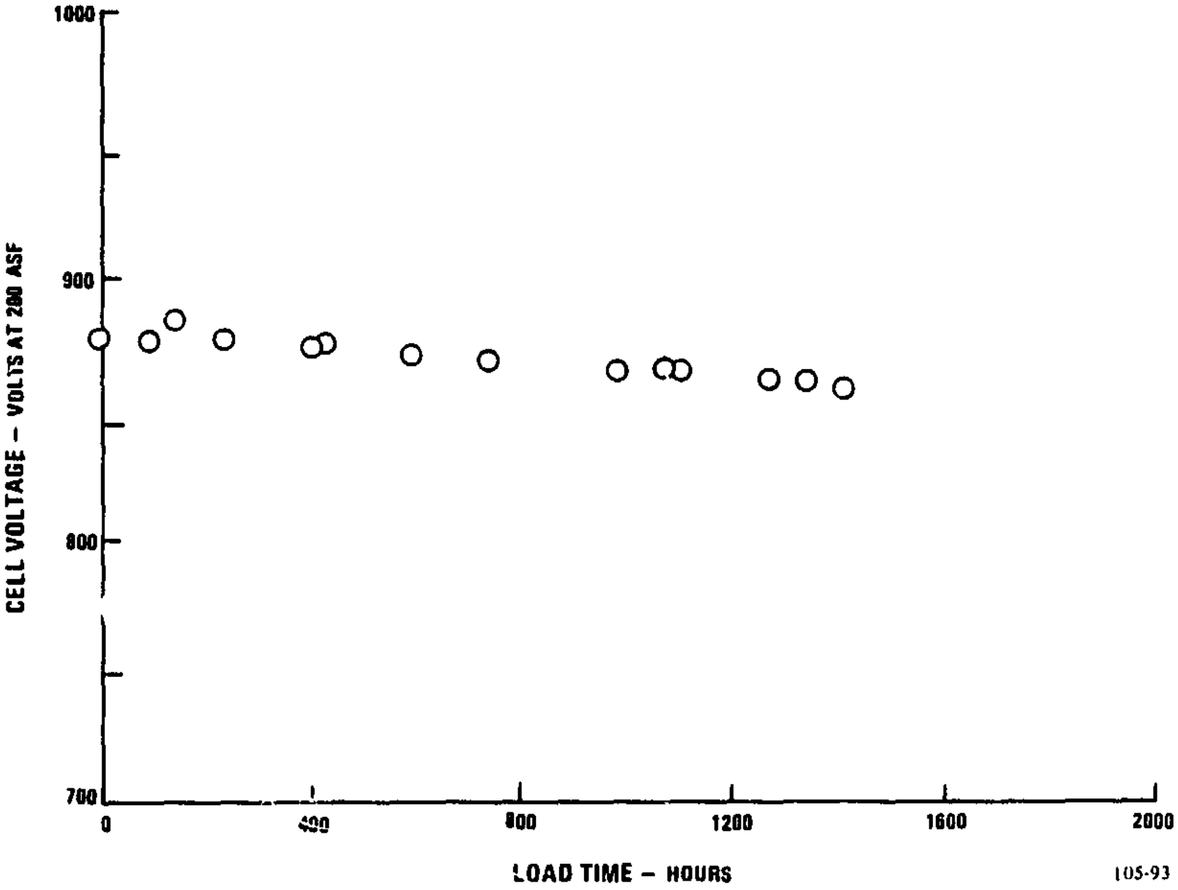


Figure 63. Research Cell (RC-90) Endurance Test
(Carbon-Ribbed Substrate Anode)

RC-92 (Bonded $LiCoO_3$ Matrix)

A matrix comprised of 96% wgt. $LiCoO_3$ 4% wgt. butyl rubber was tested in RC-92 with standard platinum-palladium catalyst anode and gold-platinum catalyst cathode. Initial performance of the cell was low due to a very high iR. After only a short period of testing reactant cross leakage developed and the test was stopped. In the forms supplied, neither $LiCoO_3$ nor $SrZrO_3$ are suitable matrix materials for use in base cells.

RC-93 (Carbon Ribbed Substrate Anode)

The cell incorporated a 10% Pt/C anode catalyst layer deposited on a nickel-treated dual porosity carbon-ribbed substrate (20W) and a standard gold-platinum catalyst cathode. The cell contains a 20-mil (.5mm) thick advanced asbestos matrix.

A total of 141-hours of testing has been completed. Cell voltage at 141-hours is 0.852V at 200 ASF (215.3 mA/cm²). The endurance test continues.

CARBON-RIBBED SUBSTRATE PERFORMANCE SUMMARY

An appreciable data base has now been developed to allow the identification of some general conclusions regarding the suitability of carbon substrates as a substitute for gold-plated screens at the anodes of hydrogen/oxygen fuel cells employing potassium hydroxide electrolyte. Significant test results from the carbon substrate evaluation are presented in Table XXVIII.

Table XXVIII. Carbon-Ribbed Substrate Performance Summary

Performance (@ 200 ASF)

Research Cell No.	Anode Catalyst	Substrate	Initial (mV)	1000 Hr (mV)	Δ (mV)
(Typical)	Pt/Pd	Screen	906	892	14
50	Pt/Pd	Single Porosity	896	882	14
79	Pt/Pd	Dual Porosity C (6W)	899	846	53
80	Pt/Pd	Dual Porosity C (20W)	906	885	21
			(902) - Average		
46	Pt/C	Single Porosity C	875	818	57
85	Pt/C	Dual Porosity C (6W)	882	849	33
86	Pt/C	Dual Porosity C (20W)	878	863	15
90	Pt/C	Dual Porosity C (20W)	878	866	12
			(878) - Average		

All of the cells had standard gold-platinum screen cathodes, therefore the differences in performance and stability are due to differences between the anode configurations.

Performance - Cells with Platinum-Palladium anode catalyst layers initially perform better than the cells with Platinum-on-Carbon catalyst layers. However, endurance testing of stacks incorporating 0.508-ft^2 (471.9 cm^2) active area Platinum-on-Carbon catalyst anode cells have demonstrated long-term performance stability. The difference in initial performance is about 24 mV at 200 ASF (215.3 mA/cm^2) and is independent of the substrate. This simply reflects the higher area of platinum in the Platinum-Palladium anodes relative to the Platinum-on-Carbon anodes, that is 10 mg Pt/cm^2 at 25 m^{2}g^{-1} versus 0.5 mg Pt/cm^2 at 100 m^{2}g^{-1}.

Stability - Approximately half of the cells lost about 14 mV during the first 1000 hours of operation. The main component of this voltage loss was an increase in anode polarization. This loss occurred with both Platinum-Palladium and Platinum-on-Carbon catalyst layers on a variety of substrates.

Cells with the 6W anode substrate showed much higher voltage loss rates. This dual porosity substrate, with dense ribs, appears to cause increasing diffusion losses with time.

The single porosity and 20W dual porosity substrates are showing promise as potential low-cost substitutes for the gold-plated screens and porous nickel ERP's used at the anode in present base cell powerplants. Additional work is needed to develop a substrate with a smaller mean pore size which will resist electrolyte expulsion during launch. Tolerance to electrolyte volume swings also needs to be demonstrated.

APPENDIX B

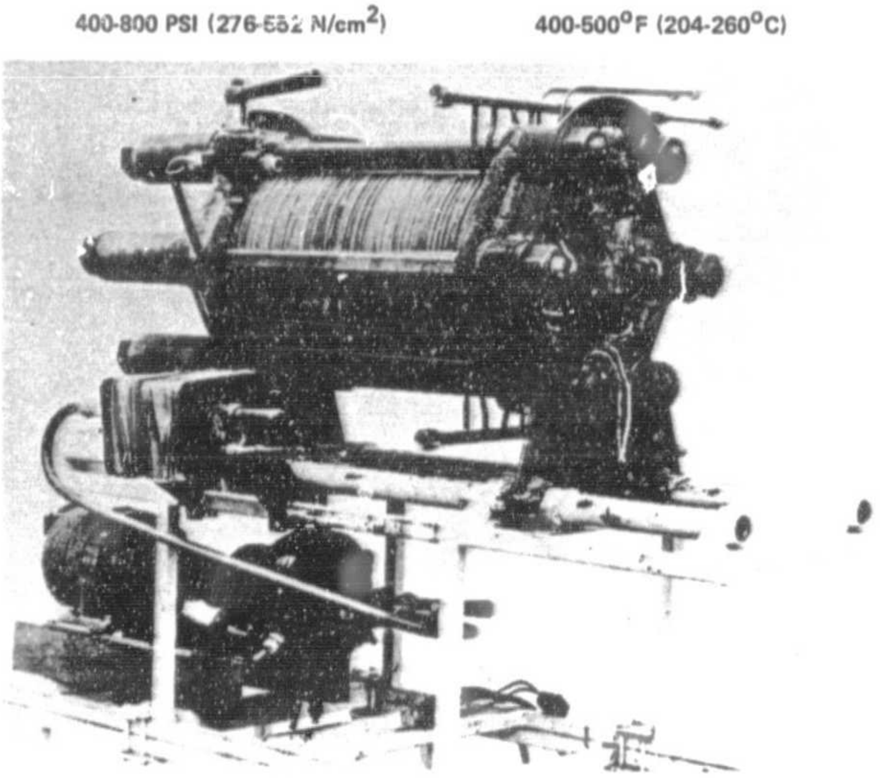
Alkaline Electrolyte Fuel Cell Development Background

United Technologies has demonstrated reduction to practice for the alkaline technology by the Apollo fuel cell powerplant and the fuel cell powerplants for the Space Shuttle Orbiter.

All of these delivery powerplants, Apollo, and Space Shuttle met firm specification requirements and operated successfully in spacecraft.

Apollo

In 1959 United Technologies ran a full-scale power section with Bacon-type cells. The test installation is shown in Figure 64.



(FC7086)

Figure 64. Bacon Fuel Cell Stack (1959)

In 1962 the first PC-3A fuel cell powerplants in flight configuration were delivered under the Apollo program. The Apollo fuel cell powerplant was qualified for manned space flight in 1965 and 92 production powerplants were delivered by 1969.

The Apollo fuel cell powerplant is shown in Figure 65. The nominal rating was 1.5 kW at 28 volts with an overload capability of 2.3 kW. The powerplant weighed 241 lbs (109 kg) and was furnished with shock mounts within the cylindrical support skirt. Three PC-3A powerplants installed in the Command and Service Module provided the primary source of electrical power for the Apollo missions.

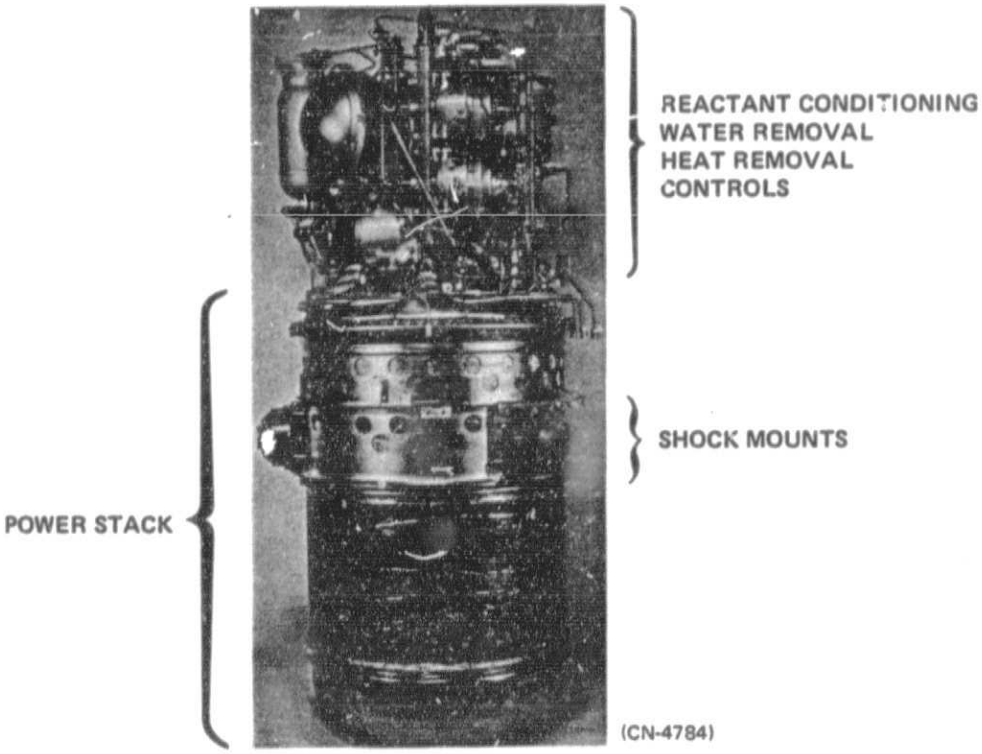
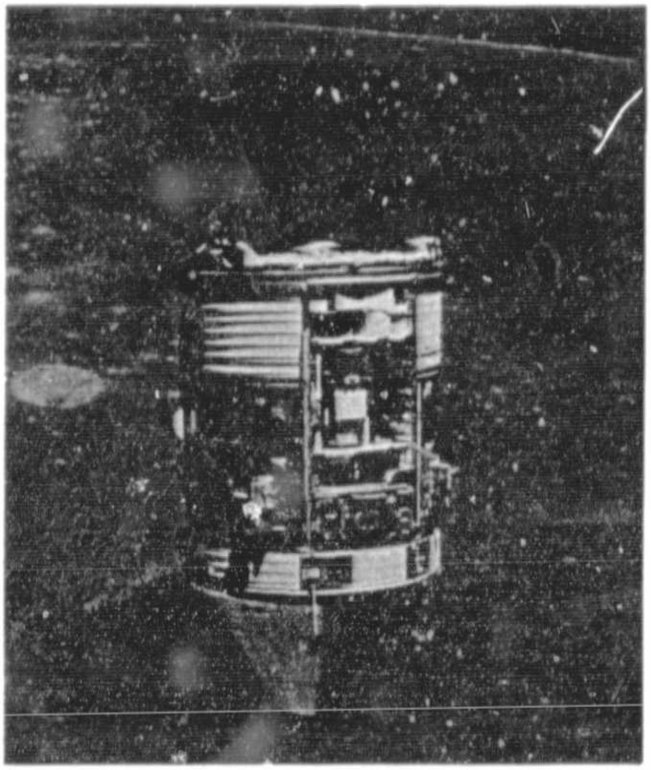


Figure 65. Apollo Fuel Cell Powerplant

The flight experience with the Apollo powerplant is summarized in Figure 66. More than 10,000 hrs of flight time were logged on 54 powerplants during 18 missions during the Apollo, Apollo-Soyuz, and Spacelab programs.



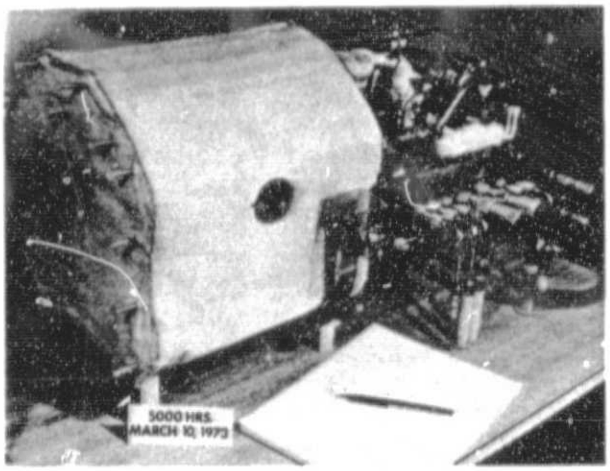
- PRIME POWER FOR COMMAND AND SERVICE MODULES
- 18 FLIGHTS INCLUDING:
  9 LUNAR/3 SKYLAB
  1 APOLLO SOYUZ
- 10, 750 HOURS OF FLIGHT TIME
- 90 POWERPLANTS DELIVERED

(WCN-1283)

Figure 66. Apollo Fuel Cell Powerplant

Space Shuttle Orbiter Powerplants

The technology for the Space Shuttle fuel cell powerplants was established by the DM-2 powerplant which was developed and demonstrated under a Technology Demonstrator program conducted for the Johnson Space Center. Figure 67 summarizes the results of a 5,000 hr test of the DM-2 powerplant at United Technologies facility. This test includes 31 simulated missions. The powerplant was shut down, cooled down, and restarted for each mission and operated to a variable load profile. The Demonstration started on August 8, 1972 and was completed in eight months on March 10, 1973. No maintenance was conducted on the powerplant during this demonstration. The powerplant was refurbished with a new power section and new bearings in the hydrogen pump and delivered to Johnson Space Center where another 5,000-hour test was completed without maintenance.



- **5072 HOUR TEST TO ORBITER LOAD PROFILE**
- **NASA-REQUIRED WATER QUALITY FOR ENTIRE TEST**
- **21 SELF-ENERGIZED STARTS**

(WCN-2194)

Figure 67. Shuttle Prototype Powerplant Endurance Test

In addition during the DM-2 powerplant program two, six-cell power sections were endurance tested, accumulating 10,000 and 10,500-hours of operation as shown on Figure 68, and a hydrogen circulation pump was tested for 10,000-hours.

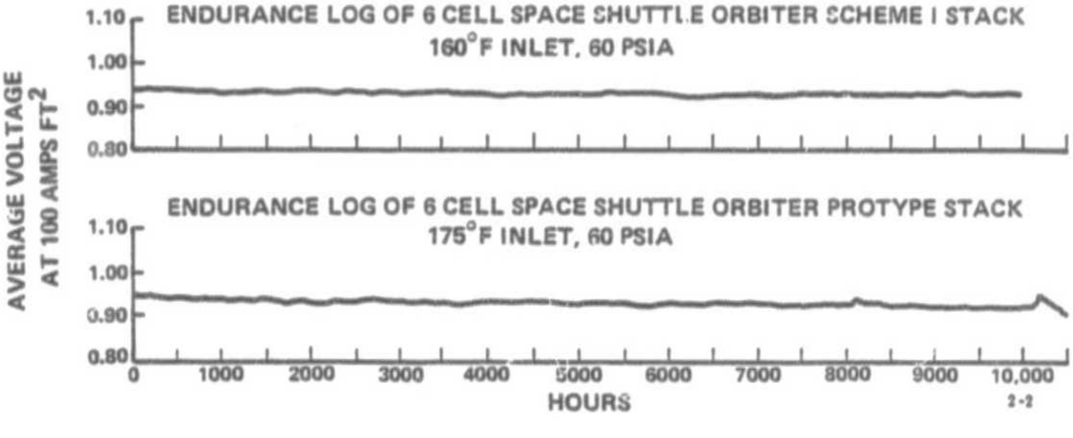
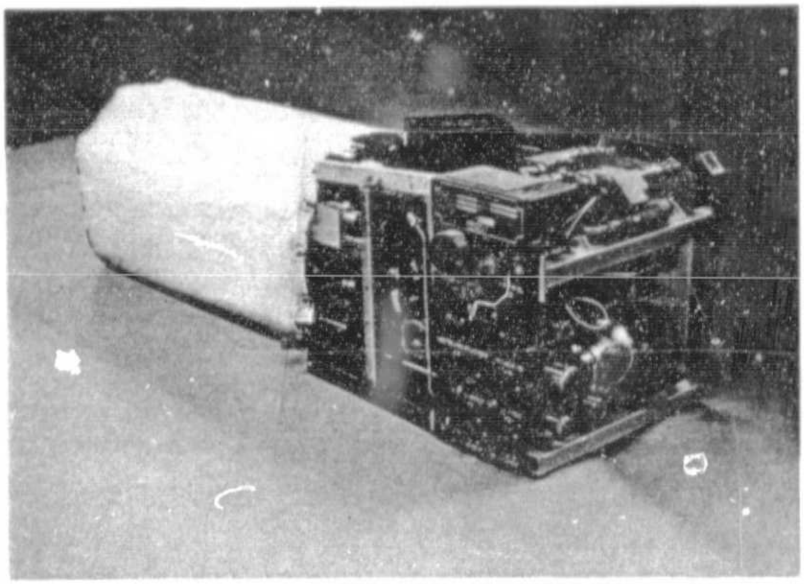


Figure 68. 10,000 Hour Power Section Tests

Three PC17 fuel cell powerplants provide the only source of electrical power on board the Space Shuttle Orbiter.

Figure 69 shows the Orbiter powerplant. The Orbiter powerplant has a nominal maximum power rating of 12 kW with an emergency overload rating of 16 kW. The Orbiter powerplant is smaller than the Apollo powerplant and delivers eight times the power. The Orbiter powerplant does not require shock or vibration isolation and is hard mounted to the vehicle structure.



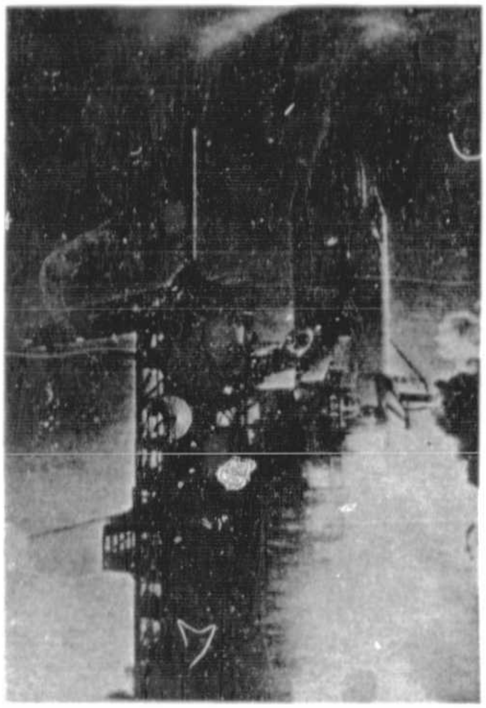
(WCN-10462)

Figure 69. Orbiter Fuel Cell Powerplant

The Orbiter fuel cell program started in January 1974. The first development powerplant test started in October 1975. Three development powerplants accumulated 8770 hours of test including accelerated vibration and operation in a simulated space vacuum.

The Orbiter fuel cell powerplant was qualified for manned space flight in June 1979. A 2000 hour qualification test including ten mission cycles and 60 start/stop cycles was completed in January 1980.

Service experience with the Orbiter fuel cell powerplant is summarized in Figure 70. A total of 6400 hours have been accumulated on fourteen powerplants installed in the Orbiter Space Crafts "Enterprise" and "Columbia" and "Challenger". The service experience includes eight flights during the Approach and landing Tests from May to October 1977 and the first Orbital flight of STS-1 in April 1981.



- 6400 HOURS ON 14 FUEL CELL POWERPLANTS
- "ENTERPRISE"
  - 8 APPROACH AND LANDING FLIGHTS
    - 212 HOURS
- "COLUMBIA"
  - 6 TEST AND OPERATIONAL FLIGHTS
    - 3473 HOURS
- "CHALLENGER"
  - 6 OPERATIONAL FLIGHTS
    - 3355 HOURS
- "DISCOVERY"
  - 2 OPERATIONAL FLIGHTS
    - 1405 HOURS

(WCN-8986)

Figure 70. Service History

In-House Demonstrator Powerplants

The PC8B series of powerplants was developed under in-house sponsored programs to improve upon the Apollo powerplant in the areas of performance, start-up characteristics, operating characteristics, endurance and powerplant weight. The PC8B powerplants are shown in Figure 71.

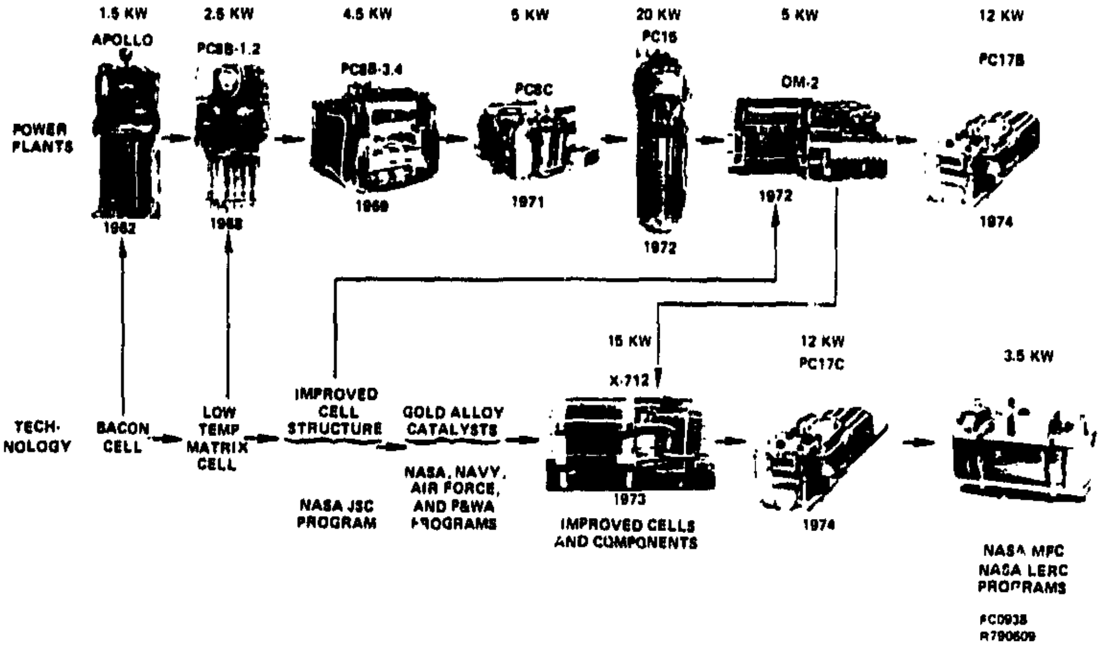


Figure 71. PC8B Demonstrator Powerplants

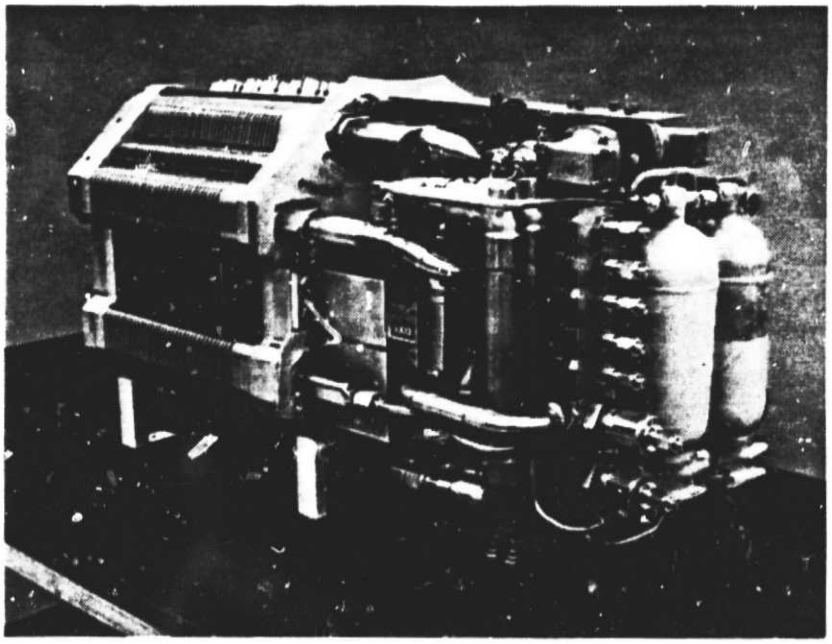
The PC8B-1 was the first powerplant incorporating low-temperature, matrix-type alkaline cells configured for a space application. Cell active area of 0.4 ft^2 (371.6 cm^2) was the same as Apollo. The PC8B-1 retained the Apollo ancillaries and mounting structure. The PC8B-2 was identical to the PC8B-1 except the interface panel and mounting structure were modified for compatibility with the Air Force Manned Orbiting Laboratory.

In 1969, the PC8B was repackaged with a stack of 0.508 ft^2 (471.9 cm^2) active area cells. Designated the PC8B-3, this powerplant was operated as an in-house demonstration unit for more than a year, accumulating 97 starts and more than 6000 hours on reactants. With an improved cooling system, its power rating was raised from 2.5 kW to 5 kW and it was designated the PC8B-4.

The 5 kW PC8C was built in 1971 with a stack of 0.508 ft^2 (471.9 cm^2) active area cells of the high power density type. This cell configuration was developed in the late 1960's in Air Force and internal research and development programs. Originally developed for operation at very high current densities, typically 3000 ASF (3229 mA/cm^2), the cell was found to have superior endurance as well. Endurance testing of this cell configuration in a National Aeronautics and Space Administration Lewis Research Center Program demonstrated over 11,000 hours of operation and a subscale laboratory cell in an internal research and development program exceeded 35,000 hours of testing. This cell configuration has been employed in all subsequent low-temperature alkaline fuel cell powerplants. The PC8C was used as an in-house demonstrator powerplant for nearly two years. During this period it accumulated 100 self-energized starts.

The X712 in-house demonstrator powerplant, Figure 72, was similar to the DM-2 powerplant but incorporated a power section of 36, 0.508 ft^2 (471.9 cm^2) active area cells with a higher performing gold-platinum cathode catalyst replacing the platinum cathode catalyst employed on the DM-2 cell.

X712 has a greater capacity coolant system than the DM-2, giving it a continuous output rating of 15 kW. X712 has been employed as a demonstrator powerplant for four years accumulating 115 self-energized starts.



(WCN-2036)

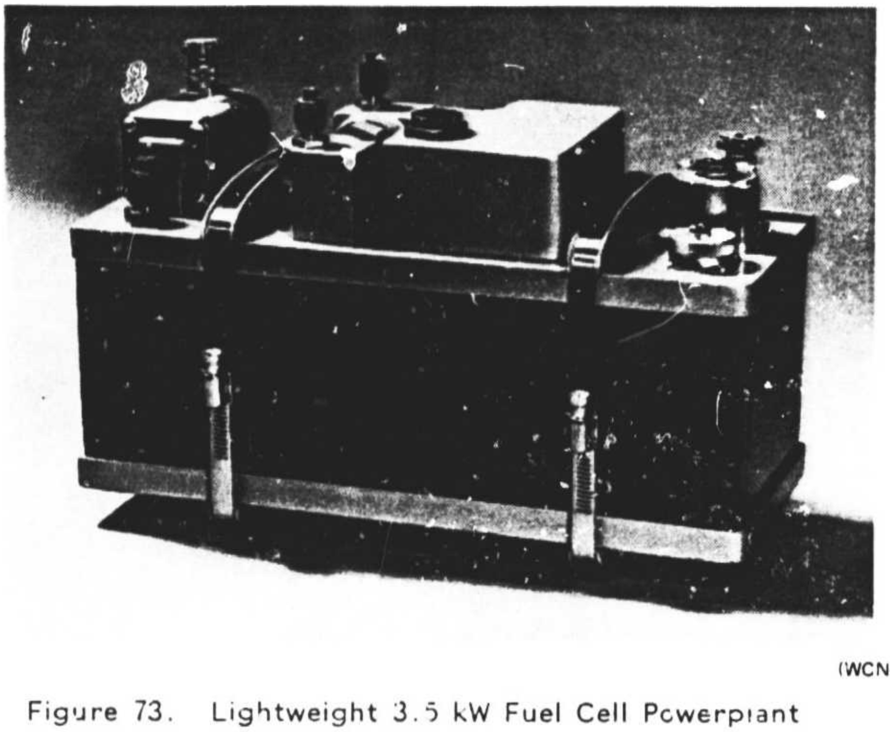
Figure 72. X712 Demonstrator Powerplant

Lightweight Fuel Cell Powerplant

A lightweight 3.5 kW fuel cell powerplant shown in Figure 73 was developed under a program sponsored by NASA-George C. Marshall Space Flight Center. The design is based upon the advanced technology lightweight fuel cell which operates with passive water removal developed under the Lewis Research Center program.

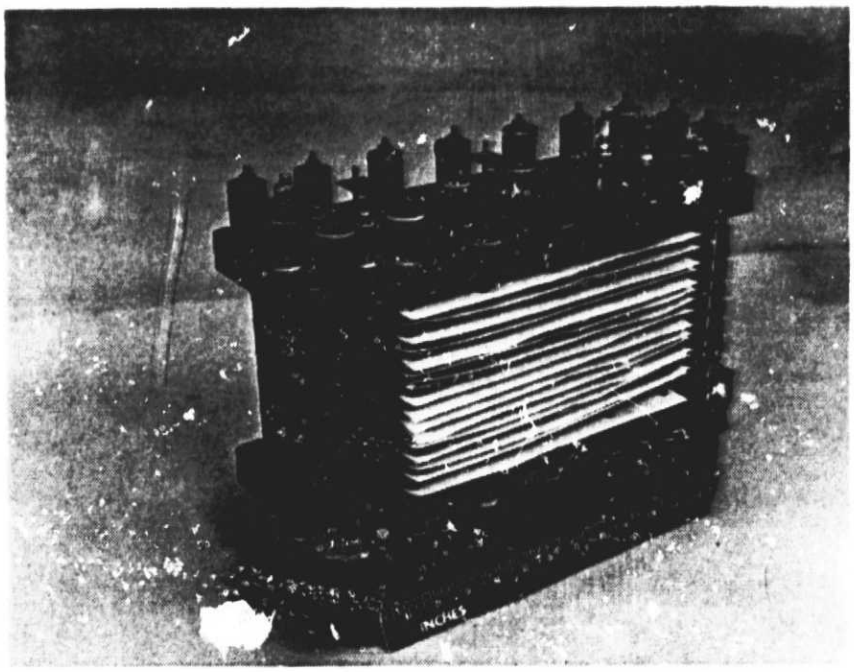
Passive water removal operation eliminates the requirement for a dynamic hydrogen pump water separator thereby allowing a powerplant design with reduced weight, lower parasite power, and a potential for higher reliability and extended endurance. The lightweight fuel cell powerplant design was based upon the requirements of advanced space missions such as Space Tug and Orbital Transfer Vehicle.

The Marshall program culminated in the fabrication of a 24-cell lightweight power section, Figure 74, which has completed a 2000-hour performance demonstration test under the Lewis Research Center Program.



(WCN-6336)

Figure 73. Lightweight 3.5 kW Fuel Cell Powerplant



(WCN-7669)

Figure 74. Lightweight Fuel Cell Power Section

Under the MSFC program, over 8,800-hours of endurance testing of two-cell modules, the basic repeating unit of the power section was completed. These tests confirmed that the lightweight cell design will:

- Satisfy the 2,500-hour voltage requirement of the Lightweight Fuel Cell Powerplant Design.
- Operate with propellent purity reactants with no significant impact upon cell performance.

A complete summary of the work completed under the Marshall Space Flight Center program is presented in reference 7.

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15. Supplementary Notes

NASA Project Manager, Mr. Dean Sheibley, Solar and Electrochemistry Division,
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16. Abstract

United Technologies Corporation has been conducting a multi-task experimental program sponsored by Lewis Research Center of NASA directed toward advancing the state-of-the-art of alkaline electrolyte fuel cells. The goal of the program was the development of an extended endurance, high performance, high efficiency fuel cell for use in a multi-hundred kilowatt Regenerative Fuel Cell for use in a future orbiting Space Station.

A design definition study of a Regenerative Fuel Cell energy storage system was conducted. The study identified system weight, volume, efficiency and an approach to system reliability. Pictorial representations of the system were prepared. The results of study are reported separately under NASA CR-174802.

This technology advancement program has identified a low-weight cell design and cell components with increased performance and endurance. Three 0.508 ft^2 (471.9cm^2) active area multi-cell stacks were assembled and endurance tested at cell temperatures between 140°F (60°C) and 180°F (82.2°C), current densities to 400 amps/ft^2 (430.6mA/cm^2) and 60 psia (41.4N/cm^2) reactant pressure. These full-size endurance tests have demonstrated (1) the long-term performance stability of the platinum-on-carbon catalyst configuration, (2) the suitability of the lightweight graphite electrolyte reservoir plate, (3) the stability of the free standing butyl bonded potassium titanate matrix structure, and (4) the long-life potential of a hybrid polysulfone cell edge frame construction.

A 18,000-hour demonstration test of multi-cell stack to a continuous cyclical load profile was conducted. A total of 12,000 cycles was completed, confirming the ability of the alkaline fuel cell to operate to a load profile simulating Regenerative Fuel Cell operation. An Orbiter production hydrogen recirculation pump employed in support of the cyclical load profile test completed 13,000-hours of maintenance free operation.

Laboratory research endurance tests at 200°F (93.3°C) and 200 amps/ft^2 (215.3 mA/cm^2) has demonstrated the suitability of the butyl bonded potassium titanate matrix, perforated nickel foil electrode substrates, and carbon-ribbed substrate anode for use in the alkaline fuel cell.

Corrosion testing of materials at 250°F (121.1°C) in 42% wgt. potassium has identified Ceria, Zirconia, Strontium Titanate, Strontium Zirconate and Lithium Cobaltate as candidate matrix materials.

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