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REPORT

SOLID POLYMER ELECTROLYTE (SPE)
FUEL CELL TECHNOLOGY PROGRAM

FINAL REPORT (PHASE 2/2A)

Contract NAS 9-14345, DRL Line Item No. 4

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

DIRECT ENERGY CONVERSION PROGRAMS
50 FORDHAM ROAD
WILMINGTON, MASSACHUSETTS 01887
ACKNOWLEDGMENTS

This report summarizes the results of a solid polymer electrolyte fuel cell technology program conducted for the National Aeronautics and Space Administration, Lyndon B. Johnson Space Center, Houston, Texas, by the General Electric Company, Direct Energy Conversion Programs, Wilmington, Massachusetts, under Contract NAS 9-14345. The period of performance was September, 1975 through November, 1976.

This work was performed under the guidance of Mr. G. D. Hydrick, Jr., Program Manager for the Power and Propulsion Branch of NASA/Lyndon B. Johnson Space Center. The overall program was directed by Mr. J. F. McElroy, Project Engineer, General Electric Company, Direct Energy Conversion Programs.
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The primary objective of this contract was to continue the evaluation of several facets of the solid polymer electrolyte fuel cell which showed promise of improving the characteristics of the technology. The major target areas were:

- Reduced Fuel Cell Costs
- Reduced Fuel Cell Weight
- Improved Fuel Cell Efficiency
- Increased Systems Compatibility

The secondary objective was to perform test evaluations of the full scale hardware which was designed and fabricated during Phase I.

The major advancement made during the Phase 2/2A program was in the ability to operate the solid polymer electrolyte cell at increased current density and at higher performance levels. Figure I displays this significant improvement in cell performance. This improved performance was obtained through a combination of increased temperature, increased reactant pressures, improved activation techniques, improved thermal control combined with the 1975 baseline cell configuration. This performance improvement has a very positive impact on the first three major target areas, (i.e., costs, weight and efficiency). The final major target area (increased systems compatibility) could also be positively effected through higher operational temperature. Since, however, it was shown that a performance optimum at less than maximum temperature exists for a given set of partial pressures and current densities, a system study would have to be conducted to determine optimum conditions and the impact on system compatibility.

Additional advances made during Phase 2/2A include:

- Demonstration of higher acid content membrane which resulted in increased performance.

- Reduced catalyst loading and low cost membrane development showed encouraging results, however, additional efforts are required in these areas.

The 0.7 Ft\(^2\) (650 cm\(^2\)) cell stack hardware fabricated during Phase I was operated with invariant performance for over 1600 hours. (See Figure 2.) This unit, which demonstrated a cell size scale up of two times the largest previous space type cell, was still operational at the end of Phase 2/2A.
Figure 1.
Figure 2. 0.7 Ft$^2$ (650 cm$^2$) Cell Stack in Test Facility
In a related development, a conductive cathode wetproofing has been demonstrated as part of the IR&D Program. This configuration will allow further cost reductions, and potentially further improve cell performance.

The main thrusts of our Phase III program recommendations are in the areas of endurance testing of proven advances and in system analysis.

The continued NASA support combined with related technology developments at GE/DECP will assure the availability of a low cost/high power density fuel cell technology within three years. This technology would then be ready for future NASA projects:

**Present Power Plant Projections $8K/KW + 4 lbs/KW.**
2.0 TECHNICAL BACKGROUND

2.1 General Background

The SPE fuel cell technology was first developed into a viable product in 1960. Over the past 16 years, improvements in both the SPE and the electrode technologies resulted in an increased field of applications to include electrolysis systems, oxygen concentrators and regenerative fuel cells. All four electrochemical devices utilize the same basic SPE and electrode components. Thus, the experience gained from one application can be used in the further improvement of the other devices.

The heart of these devices is the SPE which is approximately a 10 mil thick plastic film fabricated from ion exchange material. This currently utilized material has a structure as follows:

\[
\begin{align*}
\text{CF}_3 \\
\text{CF}_2 - \text{CF} \quad \text{CF} \\
\text{SO}_3 
\end{align*}
\]

This is essentially a sulfonated analog of Teflon with physical properties very similar to Teflon.

The use of the SPE as the sole electrolyte in an electrochemical system offers the following advantages:

- Minimum weight
- Immobile and invariant during life
- Minimum volume
- Ease of handling during assembly
- Capability of handling high pressure differentials across the membrane
- No tendency to react with CO₂ to form carbonates
2.1.1 Solid Polymer Electrolyte Technology

The Nafion® SPE described above is a product of duPont and has been utilized extensively by GE/DECP in electrochemical applications since 1968. This membrane is extremely stable, both physically and chemically, while exhibiting excellent physical and electrochemical properties. These properties are listed below:

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<td>Tensile</td>
<td>&gt; 2500 psi ( &gt; 17,235 kN/m²)</td>
</tr>
<tr>
<td>Elongation</td>
<td>&gt; 120%</td>
</tr>
<tr>
<td>Burst Strength, unsupported</td>
<td>&gt; 100 psig ( &gt; 689 kN/m² gauge)</td>
</tr>
<tr>
<td>Water Content</td>
<td>25-40 Weight %</td>
</tr>
<tr>
<td>Resistivity</td>
<td>&gt; 15 ohm-cm</td>
</tr>
<tr>
<td>Life in Electrochemical System</td>
<td>&gt; 34,000 hours demonstrated to date in fuel cells</td>
</tr>
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<td>Thermal Stability</td>
<td>&gt; 300°F ( &gt; 149°C)</td>
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The Nafion® SPE is the latest in a series of GE/DECP membrane systems that included phenol-formaldehyde sulfonic, polystyrene sulfonic, and trifluorostyrene sulfonic acids. The Nafion® SPE was the only membrane that offered the excellent combination of physical and chemical stability required in the electrochemical environments.

The first Nafion® SPE's tended to degrade slowly in an electrochemical environment as evidenced by a slow generation of HF and CO₂ from the operating device. However, this problem has been rigorously researched by both duPont and GE/DECP and has culminated in a stack (four cells) of 0.38 ft² (354 cm²) (AFC 6) fuel cells that has been operating over 39,000 hours with a projected life of over 100,000 hours. The performance characteristic over its life to date is shown in Figure 3. It is operating at 70 psia (483 kN/m²) oxygen-side pressure and has been run at 180°F for over 31,000 hours with current densities up to 260 ASF (.28 amps/cm²).

2.1.2 Electrode Technology

The GE/DECP electrode structures are thin catalyst layers pressed onto the SPE surface. The catalyst/SPE electrode also contains a thin (3-4 mil)
4-CELL BUILDUP

COOLANT TEMP.: 180°F EXCEPT AS NOTED
O₂ PRESSURE: 70 psia
H₂ PRESSURE: 65 psia

Fuel Cell Buildup (AFC 6) Life Test Performance History

Figure 3.
(0.0762–1.016 mm) current collector screen, and in order to prevent water masking of the fuel cell oxygen electrode where product water is formed, a wetproofing film is placed on top of the catalyst/current collector. The performance of these electrode structures has been invariant for over 39,000 hours with no evidence of performance decay.

2.2 Major Fuel Cell Programs

The Gemini spacecraft program marked the first operational use of an SPE unit. The General Electric solid polymer electrolyte fuel cells used on that program successfully completed all seven spacecraft missions, accumulating a total of 850 hours (5000 stack-hours) of flight operation with an excellent record of performance and reliability. The spacecraft system included two 1-KW modules, each containing three 32-cell stacks.

A total of 250 stacks of the production configuration were built during the Gemini program. Most of these were used for reliability, endurance, and over-stress testing and accumulated more than 80,000 stack-hours of operating experience in addition to flight operation.

A second-generation model of this fuel cell design successfully operated in orbit continuously for over 40 days (planned mission of 30 days) on Biosatellite Spacecraft 501 and stopped functioning only when the reactants were depleted.

The so-called "back-to-back" cell design concept, where cathodes of adjacent cells faced each other, was initially developed for Air Force satellites. This concept was continued under NASA's sponsorship.

In this NASA Space Shuttle Technology Development Program, General Electric fabricated and evaluated flightweight fuel cell hardware. Noteworthy is the fact that a 3-cell assembly completed over 6500 hours of life testing, a 38-cell stack accumulated 5000 hours, and a 32-cell stack with ancillary components in a prototype flight configuration achieved 2000 hours of operation.

The technology developed during the Space Shuttle technology has recently been repackaged as a 3 KW fuel cell for the Navy's High Altitude Super-Pressurized Powered Aerostat (HASPA).

Recently under sponsorship of NASA's Lewis Research Center, initial development of a new SPE concept was started. The major feature of this concept was the removal of product water via a dynamic recycling oxygen system as opposed to the traditional wick and separator approach used since the Gemini Program. The advantages of this concept are in the areas of weight, cost and efficiency.
Figures 4 through 6 display the various products described above. Figure 7 is a graphical presentation of the past 15 years of progress in the SPE fuel cell technology.
SPACE SHUTTLE FUEL CELL PROTOTYPE

- 5000 HOURS LIFE TEST - 2.5 KW STACK
- 2000 HOURS LIFE TEST - 2.5 KW MODULE

GEMINI FUEL CELL

- FIRST FUEL CELL SYSTEM IN SPACE
- SEVEN SUCCESSFUL MANNED SPACE FLIGHTS

BIOSATELLITE FUEL CELL

- OPERATED 40 DAYS IN SPACE
- FIRST APPLICATION OF FLUOROCARBON SPE

Figure 4.
Figure 5. Fuel Cell Module FS-2 (3KW)
Figure 6. Complete Fuel Cell Power System, Including Cryogenic Tasks, in the Process of Assembly for U.S. Navy Balloon Program
Figure 7.

Progress of SPE Space Fuel Cell Technology
3.0 TECHNOLOGY ACTIVITIES

The major tasks of this Phase 2/2A program were twofold. The first task was to continue to evaluate several promising aspects of the solid polymer electrolyte fuel cell in laboratory-sized hardware. The second major task was to perform operational tests on the full scale hardware fabricated during Phase I. Figure 8 shows the overall Phase 2/2A program milestones.

3.1 Laboratory Evaluations

This major task was subdivided into three major sub-tasks as follows:

1) Temperature/pressure performance
2) Catalyst Performance
3) Materials Performance

In order to determine the performance of a standard cell, a baseline configuration was established utilizing the 1975 current state-of-the-art. This 1975 baseline consisted of:

- Solid Polymer Electrolyte
  - duPont's Nafion®
  - 5 mil (.1270 mm) thick
  - 35-40% H₂O
  - 1200 equivalent weight
  - Platinized

- Anode Electrode
  - 4 grams/Ft² (4.3 mg/cm²)
  - 15% T-42 (Teflon)
  - 64% Pt, 21% Ru, 15% WO₂
  - Gold screen distribution assembly

- Cathode Electrode
  - 4 grams/Ft² (4.3 mg/cm²)
  - 12.5% T-30
  - 100% Pt black
  - 1/4 inch (6.35 mm) stand pipe
  - 3 mil (.0762 mm) niobium screen
**Milestone Chart**

<table>
<thead>
<tr>
<th>Item</th>
<th>1975</th>
<th>1976</th>
<th>1977</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>S O N D</td>
<td>J F M A M J J A S O N D</td>
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<td>1.0</td>
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<td>1.1</td>
<td>Temperature/Pressure</td>
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<td>1.2</td>
<td>Catalyst</td>
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<tr>
<td>1.3</td>
<td>Materials</td>
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<td></td>
<td>Chemplast</td>
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<td>High IEC</td>
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<td>Low Cost Membrane</td>
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<td>Current Collectors</td>
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<td>(Inc. H2 Distribution)</td>
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<tr>
<td>2.0</td>
<td>Stack NT-1 Evaluation</td>
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<td>2.1</td>
<td>Facility Preparations</td>
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<td>2.2</td>
<td>Facility/Checkout</td>
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</tr>
<tr>
<td>2.3</td>
<td>Test Evaluation</td>
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<tr>
<td></td>
<td>Final Report - Volume II</td>
<td>🔄</td>
<td>🔄</td>
</tr>
</tbody>
</table>

*Figure 8. NASA/JSC Technology Program Phase II/IIA*
Current Collection

BiPolar

Open cathode gap

The baseline configuration was sized to fit the laboratory 3 x 3 inch hardware shown on Figure 9. The baseline cell performance was determined when operating on oxygen/hydrogen, air/hydrogen, and oxygen/contaminated hydrogen. These performance results are displayed on Figure 10. The performance displayed on Figure 10 will be utilized throughout this report to be compared with the performances of the new configuration variations.

3.1.1 Temperature/Pressure Performance

The purpose of this sub-task was to evaluate variations of the 1975 baseline cell configuration at higher temperature and pressure conditions. Work performed during Phase IA indicated that significant performance improvement and improved system compatibility could be obtained at higher temperatures and pressures. A summary of the results of work performed under this task is as follows:

- With modifications to the test fixture thermal configuration, stable current densities up to 1500 ASF were obtained. This represented a three-fold increase in current density capability at a fixed cell voltage.

- Performance and chemical stability were obtained at high temperature and pressure by facility material changes and proper reactant humidification.

- Cell performance was characterized vs. temperature, pressure, and current density. This characterization showed that optimum performance was influenced by each of the variables.

- The corrective action of increased humidification of reactant to chemically stabilize the cell resulted in an occasional water slug introduction into the anode chamber causing a temporary performance fall off. Although this condition is unlikely on full scale hardware with the very accurate end plate humidifier developed during the space shuttle technology program, this condition must be corrected in the laboratory hardware in order to obtain accurate performance data at temperatures approaching 300°F. Fixture design modifications have been introduced and initial test results indicate that the water slug condition has been minimized if not eliminated.
Figure 9. 3" x 3" Fuel Cell Hardware
Figure 10. Baseline Cell Performance
A total of six cells were fabricated and performance tested as part of this sub-task. The major variables in the six configurations involved the test fixture and the current collector/gas distribution design. Table I describes the variations from baseline configuration for all six test samples. Also shown on Table is a performance comparison to the 1975 baseline performance. Facility modification to allow high temperature, pressure and current density were made a few months after the start of Phase 2, which prohibited a direct performance comparison to baseline because of an inability to operate at the very low baseline pressures (i.e., 16 psia cathode and 15 psia anode).

3.1.1.1 Cell NT(II)-1-1 (10 mil SPE-6/0 Anode Screen)

The thicker membrane was selected for this first high tempered/high pressure cell to allow for greater differential pressures at high temperatures. The 6/0 anode screen also gave greater support to the cell than the standard 3/0 screen.

Figure 11 displays the cell performance at various temperatures and pressures while operating on hydrogen and oxygen. Figure 12 shows the cell performance on air/hydrogen at three different pressure levels. Testing was terminated when attempted operation at 220°F and 45 psig resulted in lower performance even though internal resistance decreased. Later, this lower performance was analyzed to be the natural result of the lower oxygen partial pressure at the increased temperature.

3.1.1.2 Cell NT(II)-1-2 (10 mil SPE-6/0 Anode Screen)

Cell NT(II)-1-2 was fabricated because at first analysis there appeared to be some defect with cell NT(II)-1-1, (i.e., lower performance at 220°F). The configuration of cell NT(II)-1-2 was identical to cell NT(II)-1-1. Figure 13 displays the cell performance at various pressure levels while at 165°F. This performance was very similar to that obtained on cell NT(II)-1-1. The pressures investigated on cell NT(II)-1-2 included a range up to 85 psig compared to a maximum of 45 psig on cell NT(II)-1-1.

The cell temperature was increased to 220°F while at 85 psig and the internal resistance decreased. Figure 14 shows the 220°F performance which was about 0.02 volt higher at 200 ASF than the 165°F performance. An additional increase in cell temperature to 250°F resulted in a further reduction in the internal impedance. The 250°F performance, also shown on Figure 14, was 0.01 volt lower at 200 ASF than the performance at 220°F. This reduction in performance was analyzed to be caused by reduced partial pressure of oxygen at 250°F. Because of the lower operating pressure of NT(II)-1-1, the performance drop off occurred at a lower temperature.
**TASK 1.1 TEMPERATURE AND PRESSURE PERFORMANCE**

**PURPOSE:**

TO EVALUATE VARIATIONS OF THE 1975 BASELINE CELL CONFIGURATION AT HIGHER TEMPERATURE AND PRESSURE CONDITIONS.

A TOTAL OF SIX CELLS HAVE BEEN EVALUATED UNDER THIS TASK.

---

### TABLE I

<table>
<thead>
<tr>
<th>Fuel Cell Number</th>
<th>Deviation from 1975 Baseline Fuel Cell</th>
<th>Performance Variance from 1975 Baseline Fuel Cell</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>[O_2/H_2] [100 \text{ ASF} ] [200 \text{ ASF}]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>165°F VDC</td>
</tr>
<tr>
<td>NT(II)-I-1</td>
<td>10 MIL SPE 6/0-3/0 Nob. Anode Screen Assembly</td>
<td>-0.004</td>
</tr>
<tr>
<td>NT(II)-I-2</td>
<td>10 MIL SPE 6/0-3/0 Nob. Anode Screen Assembly</td>
<td>-</td>
</tr>
<tr>
<td>NT(II)-I-3</td>
<td>1975 Baseline in High Current Density Fixture</td>
<td>HIGH PRESSURE FACILITY UNABLE TO OPERATE AT NEAR AMBIENT PRESSURES FOR DIRECT PERFORMANCE COMPARISON</td>
</tr>
<tr>
<td>NT(II)-I-4</td>
<td>1975 Baseline with Gold Cathode Screen in High Current Density Fixture</td>
<td>HIGH PRESSURE FACILITY UNABLE TO OPERATE AT NEAR AMBIENT PRESSURES FOR DIRECT PERFORMANCE COMPARISON</td>
</tr>
<tr>
<td>NT(II)-I-5</td>
<td>1975 Baseline in High Current Density Fixture</td>
<td>HIGH PRESSURE FACILITY UNABLE TO OPERATE AT NEAR AMBIENT PRESSURES FOR DIRECT PERFORMANCE COMPARISON</td>
</tr>
<tr>
<td>NT(II)-I-6</td>
<td>1975 Baseline with gold cathode screen in high current density fixture with built in Hg side trap and no O2 side standpipe.</td>
<td>HIGH PRESSURE FACILITY UNABLE TO OPERATE AT NEAR AMBIENT PRESSURES FOR DIRECT PERFORMANCE COMPARISON</td>
</tr>
</tbody>
</table>
Figure 11.

Fuel Cell NT/T-1-1 (B/A Anode - 10 Mil)

- Cs/H2 (120°F) R = 0.0083 Ohm O2 Press. - 37° H2O ΔP = 17° H2O
- Cs/H2 (155°F) R = 0.0089 Ohm O2 Press. - 37° H2O ΔP = 17° H2O
- Cs/H2 (185°F) R = 0.0066 Ohm O2 Press. - 15 Psig ΔP = 17° H2O
- Cs/H2 (185°F) R = 0.0069 Ohm O2 Press. - 15 Psig ΔP = 90° H2O
- Cs/H2 (185°F) R = 0.0107 Ohm O2 Press. - 30 Psig ΔP = 15° H2O
- Cs/H2 (185°F) R = 0.0068 Ohm O2 Press. - 30 Psig ΔP = 90° H2O
- Cs/H2 (185°F) R = 0.010 Ohm O2 Press. - 45 Psig ΔP = 17° H2O
- Cs/H2 (185°F) R = 0.0068 Ohm O2 Press. - 45 Psig ΔP = 90° H2O

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Figure 12.

Fuel Cell NT(0)-1-1 (O/A Anode - 10 Mil)

- Air/H₂ (165°F) R = 0.0104 Chm Air Press. - 37" H₂O ΔP = 17" H₂O
  2.5 x Stoich Air
- Air/H₂ (165°F) R = 0.0111 Chm Air Press. - 15 Psi ΔP = 17" H₂O
  2.5 x Stoich Air
- Air/H₂ (165°F) R = 0.0069 Chm Air Press. - 15 Psi ΔP = 90" H₂O
  2.5 x Stoich Air
- Air/H₂ (165°F) R = 0.0069 Chm Air Press. - 45 Psi ΔP = 90" H₂O
  2.5 x Stoich Air

ORIGINAL PAGE IS
OF POOR QUALITY
Figure 13.
Figure 14.

Fuel Cell NT/TE-1-2 (5/0 Anodes -19 Min)

- $O_2/H_2 (220^\circ F)$ R=0.0061 Ohm, $O_2$ Pressure 85 psig, ΔP=90"

- $O_2/H_2 (250^\circ F)$ R=0.0039 Ohm, $O_2$ Pressure 85 psig, ΔP=90"

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The cell temperature was slowly increased to 300°F. Cell performance fell as the temperature increased until at 300°F a loss of 0.08 volt at 100 ASF from the 250°F performance was observed. The pressure level was increased to 100 psig but this had only a slight impact on the performance. Cell temperature was reduced while maintaining 100 psig and 90"H2O Δp at 100 ASF. Figure 15 shows the performance vs. temperature of cell NT(II)-1-2 as the temperature was decreased with the normal anode side coolant inlet. Figure 15 also shows the temperature vs. performance results when the coolant flow was reversed to cathode side coolant inlet.

When the coolant temperature was reduced to 250°F the operational pressure level was reduced from 100 psig to 63 psig. This reduction produced the same partial pressures of the reactants at 250°F as had been present at 300°F with a total pressure of 100 psig. The performance at 100 ASF at this level was .866 volt which was slightly less than the just completed 300°F/100 psig performance at 100 ASF of .870 volt.

Cell NT(II)-1-2 was then operated with a strong wetting delta temperature, (i.e., the cathode coolant plate was maintained a minimum of 15°F above the anode coolant temperature). Starting at an anode temperature of 720°F the temperatures were slowly raised to 300°F anode temperature. With the load held constant at 100 ASF, the ΔP held at 90"H2O and the total oxygen side pressure maintained at 100 psig. An optimum performance was once again displayed at approximately 240°F (see Figure 16). By the time the anode coolant had reached 300°F, cell performance had fallen 0.038 volt at 100 ASF from the optimum 240°F performance.

Steady state operation with the cathode at 318°F and the anode at 300°F resulted in an additional decay of 0.017 volt at which time stable performance was obtained. With the 18°F wetting ΔT, it did not seem possible that the cell was suffering from local evaporative drying. In order to assure that this was the case, the wetting ΔT was increased to 42°F. As can be observed on Figure 17 no performance improvement, in fact a slight voltage reduction occurred, during this period of increasing the ΔT in the wetting direction.

The cathode temperature was then slowly reduced while maintaining the anode temperature at a constant 300°F. Cell performance optimized in the cathode temperature range between 307°F and 304°F. (See Figure 17). At 300°F cathode temperature (0.0°F ΔT between anode and cathode coolant) performance was only off optimum by 0.002 volt. As the cathode temperature was further reduced into the drying ΔT region, a more rapid performance loss was detected. (Also shown on Figure 17).
Cell NT (II) - 1 - 2

- Decreasing Temperature Anode Coolant Feed
- Increasing Temperature Cathode Coolant Feed
- Decreasing Temperature Cathode Coolant Feed

Figure 15.
Figure 16.

Cell Voltage at 100 AEC - 100 psig and 90°F H₂O ΔP

Temperature °F (Anode)

(263°F) (273°F) (277°F) (284°F) (286°F) (297°F) (306°F) (313°F) (318°F)

(•) - Cathode Coolant Temperature

Cell NT(II)-1-2
Cell Voltage at 100 ASF - 100 psig and 90°, H₂O Δp

Anode Temperature - 300°F

Start

End of Test

Temperature °F (Cathode)

Cell NT(II)-1-2

Figure 17.
In summary, the testing of cell NT(II)-1-2 and in retrospect that of cell NT(II)-1-1 showed the major influence of partial pressure on output performance at the higher temperatures. It was also shown that differential temperatures had only a minor effect on cell output performance.

3.1.1.3. **Cell NT(II)-1-3 (High Current Density Fixture)**

Cell NT(II)-1-3, a 1975 baseline configuration, was installed into a new high current density test fixture and installed on the high temperature/high pressure test facility. The purpose of this cell evaluation was two-fold. Firstly, it was intended to demonstrate improved cell performance at higher temperature, pressure and current density with the improved thermal characteristics of the new fixture and secondly, it was intended to compare the performance decay and high HF release observed on the low catalyst loading cells with a baseline cell. (Paragraph 3.1.2 will cover the performance of low catalyst loading cells.)

The activated cell NT(II)-1-3 was operated for several days at selected temperatures. Figure 18 displays the performance of the cell at each temperature. The voltage performance shown on Figure 1 represented the highest level at a given current density and the highest sustained and stable current density obtained to that point in time with the 1200 E.W. solid polymer electrolyte fuel cell, (i.e., 800 ASF at .635 volt DC).

Figure 19 displays the results of a mini-endurance test program on cell NT(II)-1-3. As can be seen on the figure, the performance remained essentially constant for the six days of operation at both 120°F and 165°F. The hydrogen fluoride release rate at these two temperatures was also within the range of expected values. Upon increasing the cell temperature to 220°F the hydrogen fluoride release rate increased over previous experience and a slow but steady performance decay was noted. During the tenth day of operation at 220°F a cross electrolyte leakage developed and the test was terminated.

A chemical analysis was performed on the cell with the following results:

- Cell water content - 42% vs. 38% initial (within measurement error)
- Milli-equivalents of H+ - .33 vs. .32 initial indicates insignificant contamination and insignificant general polymer degradation.
Figure 18.
Figure 19.

Performance and Chemical Stability
The following visual observations were also made on cell NT(II)-1-3:

- The cathode niobium screen, when peeled from the electrolyte displayed some multicolored oxidation near the reactant inlets. This may have been related to the heat release during the cross electrolyte occurrence.

- Following electrode stripping, the hydrogen inlet area of the polymer was found to have undergone degradation/delamination. This undoubtedly resulted from subsaturation of the hydrogen fuel.

In summary cell NT(II)-1-3 displayed performance levels and stable current densities considerably higher than had been previously demonstrated. The cell teardown observations indicated that subsaturated hydrogen fuel was a major contributor to the performance and chemical instability.

3.1.1.4 Cell NT(II)-1-4 (Gold Cathode Screen - New Fixture)

Cell NT(II)-1-4 was fabricated with a gold cathode screen in place of the niobium screen utilized on cell NT(II)-1-3. The oxidized niobium screen observed in the teardown of cell NT(II)-1-3 was thought to have possibly contributed to the performance decay of that cell. Prior to activation, rust was noted in the 316 stainless steel reactant inlet lines. These lines were replaced with titanium lines.

Cell NT(II)-1-4 was activated and the reactant humidifiers were maintained 10°F above the coolant temperature in order to assure complete reactant saturation at the coolant temperature.

Figure 20 displays the performance accumulated during a mini endurance run with the following items of interest:

- Unlike cell NT(II)-1-3, cell NT(II)-1-4 showed stable performance with time at 260°F.

- Unlike cell NT(II)-1-3, cell NT(II)-1-4 showed normal HF release rates.

The improved performance stability and lower HF release probably was the result of one or a combination of the three corrective actions taken on this cell:

- Gold Screen Cathode
- Titanium Reactant Inlet Tubes
- Humidifier Temperatures 10°F above Cell Temperature.

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Figure 20. Performance and Chemical Stability

Fuel Cell NT(II)-1-4
115 psia Oxygen
90° H₂O O₂/H₂ ΔP

○ DC Volts @ 300 ASF
□ IR Free Volts @ 300 ASF
△ HF in Product H₂O
The cell temperature was increased to 300°F, however, an automatic shutdown occurred during the first night of operation which appeared to have resulted from excess hydrogen-side water. A polarization curve was taken at the end of each temperature stabilization run and is shown on Figure 21. The significant data points include:

<table>
<thead>
<tr>
<th>Cell DC Voltage At</th>
<th>100 ASF</th>
<th>300 ASF</th>
<th>1000 ASF</th>
</tr>
</thead>
<tbody>
<tr>
<td>165°F</td>
<td>.899</td>
<td>.814</td>
<td>.634</td>
</tr>
<tr>
<td>220°F</td>
<td>.918</td>
<td>.842</td>
<td>.702</td>
</tr>
<tr>
<td>260°F</td>
<td>.920</td>
<td>.846</td>
<td>.720</td>
</tr>
</tbody>
</table>

A complete non-operational checkout of cell NT(II)-1-4 showed that no detectable damage to the cell had occurred as a result of the shutdown. The cell was reactivated and the original performance on oxygen and hydrogen was obtained. The cathode chamber was switched from oxygen to air and a series of polarizations were performed at various operational temperatures. Figures 22 and 23 reflect these results. The significant data points include:

<table>
<thead>
<tr>
<th>Cell DC Voltage At</th>
<th>100 ASF</th>
<th>300 ASF</th>
<th>700 ASF</th>
</tr>
</thead>
<tbody>
<tr>
<td>120°F</td>
<td>.842</td>
<td>.735</td>
<td>-</td>
</tr>
<tr>
<td>165°F</td>
<td>.865</td>
<td>.780</td>
<td>.620</td>
</tr>
<tr>
<td>220°F</td>
<td>.890</td>
<td>.810</td>
<td>.675</td>
</tr>
<tr>
<td>260°F</td>
<td>.890</td>
<td>.815</td>
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</tr>
<tr>
<td>300°F</td>
<td>.863</td>
<td>.783</td>
<td>-</td>
</tr>
</tbody>
</table>

Following the air/hydrogen operation repeat testing on oxygen/hydrogen was performed with polarization curves shown on Figure 24. Some performance improvement over initial results were obtained with the following significant data points:

<table>
<thead>
<tr>
<th>Cell DC Voltage At</th>
<th>100 ASF</th>
<th>300 ASF</th>
<th>1000 ASF</th>
<th>1500 ASF</th>
<th>1600 ASF</th>
</tr>
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<tbody>
<tr>
<td>120°F</td>
<td>.905</td>
<td>.820</td>
<td>.675</td>
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<tr>
<td>165°F</td>
<td>.915</td>
<td>.840</td>
<td>.710</td>
<td>.612</td>
<td>-</td>
</tr>
<tr>
<td>220°F</td>
<td>.940</td>
<td>.875</td>
<td>.740</td>
<td>.635</td>
<td>.600</td>
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<tr>
<td>260°F</td>
<td>.950</td>
<td>.880</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>300°F</td>
<td>.915</td>
<td>.850</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Fuel Cell NT(II)-1-4
Cathode - 115 Psia
Anode - 112 Psia

O\textsubscript{2}/H\textsubscript{2} Performance vs. Temperature

Figure 21.
Figure 22.

Cell Voltage - VDC

Air/H₂ Performance vs. Temperature

Fuel Cell NT(III)-L-4
Anode - 112 Psia
Air Flow - 2.5 L/Min.

Air/H₂ @ 300°F
Air/H₂ @ 220°F
Air/H₂ @ 165°F
Air/H₂ @ 120°F

Current - ASF
Fuel Cell NT(II)-1-4
Cathode - 115 Psia
Anode - 112 Psia
Air Flow - 2.5 L/Min.

Air/H_2 @ 220°F
Air/H_2 @ 165°F

Air/H_2 - High Current Density Performance
Figure 23.
O₂/H₂ - High Current Density Performance

Figure 24.
Figure 25 presents the power voltage relationship of the post air operation of cell NT(II)-1-4. This shows a peak power projection of approximately 1100 watts per ft² (1.2 watts/cm²).

Additional testing was conducted on cell NT(II)-1-4 to investigate cell performance vs. oxygen partial pressure and to further identify limiting current densities. Figure 26 displays cell performance at 120°F and three pressure levels. As can be seen in Figure 26, performance significantly increased as did the "limiting" current with increased pressures. The "limiting" current was identified as the point where cell voltage started to become unstable.

Figures 27 and 28 show cell NT(II)-1-4 performance at 165°F and 220°F respectively at the three selected partial pressure levels. As the cell temperature was increased from 120°F to 165°F and then to 220°F, cell performance at the lower current densities increased. However, the "limiting" current and the high current density performance decreased. Figure 29 displays the performance relationship between temperature, oxygen partial pressure and current density.

Several conclusions/observations can be made relative to the cell NT(II)-1-4 testing:

- Performance stability demonstrated up to and including 260°F operation.
- Chemical stability demonstrated up to and including 260°F operation.
- Stable cell voltage up to 1600 ASF demonstrated.
- Optimum cell performance at a total pressure of 115 psia appears to be in the 220-260°F range.
- Some downward bend in the polarization curve noted at current densities over 1000 ASF. This is especially true at the higher temperatures (i.e., 260°F and 300°F).
- Higher air "stoichi" required at higher pressures indicating less than optimum cathode distribution configuration.
- "Limiting" current decreases with increasing temperatures.
- Performance and "limiting" current increase with increasing partial pressure.
Projected Power

Voltage

Projected Voltage

Cell NT (II)-1-4
220°F
115 Psia
90" H2O ΔP

O2/H2 - Power vs. Voltage

Figure 25.
Performance vs. Pressure

Figure 26.
Figure 27. Performance vs. Pressure

Fuel Cell NT(II)-1-4
165°F
O₂/H₂ ΔP - 90" H₂O

O₂ Partial Pressure
- 1 Atmos.
- 2 Atmos.
- 3 Atmos.
Figure 28.

Performance vs. Pressure

Fuel Cell NT(II)-1-4
220°F
O₂/H₂ ΔP = 90° H₂O

O₂ Partial Pressure
- 1 atmos.
- 2 atmos.
- 3 atmos.

Current - ASF

Cell Voltage - VDC
Performance vs. Pressure, Temperature and Current Density

Figure 29.
At a fixed reactant partial pressure, increased current densities obtain optimum performance at lower cell temperatures.

3.1.5 Cell NT(II)-1-5 (High Current Density Fixture)

Cell NT(II)-1-5 was a baseline cell installed in the high current density fixture. The main purpose of this cell test was to demonstrate that a niobium cathode screen does not contribute to performance or chemical decay. Since niobium replacement with gold screen was one of the three corrective actions taken on cell NT(II)-1-4, it was advantageous for obvious cost reasons to eliminate niobium screen as a contributor to performance decay.

Cell NT(II)-1-5 was operated for 26 days showing low HF release rates and stable performance throughout. (Figure 30 shows the performance results.)

3.1.6 Cell NT(II)-1-6 (Gold Screen Cathode - H₂O Trap)

Cell NT(II)-1-6 was assembled into a modified high current density fixture containing a built in H₂O trap on the anode side. The modification was added to eliminate the periodic performance losses due to water slug introduction into the anode chamber. Figure 31 displays the high current density performance of cell NT(II)-1-6 at 165°F. Two items of significance were noted during this 165°F run. First, no evidence of excess anode side water was observed and second, the polarization curve had no slight downward bend after exceeding the 1000 ASF level.

3.1.2 Catalyst Reduction Performance

It was the purpose of this subtask to demonstrate techniques of lowering catalyst noble metal content without loss of output performance. Work performed during Phase I was unsuccessful in producing a cell configuration that provided equivalent performance with lower catalyst loadings. However, techniques were identified which indicated promise of producing the desired results. A summary of the results of work performed under this subtask is as follows:

- Boron carbide was identified in technical reports as a high performance catalyst extender when utilized with a liquid electrolyte cell. Various attempts at using boron carbide with a liquid electrolyte were unsuccessful and effort to optimize the boron carbide material were discontinued.

- Five techniques of lowering anode side noble metal content demonstrated promising initial performance. These reductions ranged from 25% to 0.5% of the baseline loading. However, all five variations suffered performance decay with endurance time.
Figure 30.

Performance and Chemical Stability

Days of Operation

Fuel Cell NT(II)-1-5
115 Ptsa Oxygen
90° H₂O O₂/H₂ P

O₂ Facility Failure
Facility Power Failure

DC Volts @ 300 ASF
IR Free Volts @ 300 ASF
H₂ in Product H₂O

Produced by long-run, high-speed desktop photocopier
Figure 31.

Cell NT(H)-1-6 (O\textsubscript{2}-H\textsubscript{2})
100 Psig Cathode
90° H\textsubscript{2}O ΔP
165°F

O\textsubscript{2}/H\textsubscript{2} - High Current Density Performance
The cause of the performance decay is under continuing investigation.

- Attempts at lowering the noble metal content on the cathode electrode were not as fruitful as the anode results, as electrode attachment was found to be poor with the reduced platinum loading.

A total of eight cells were fabricated as part of this subtask. Meaningful test results were obtained from only six as two cells failed to perform due to physical detachment of the cathode electrode. Table II describes the variations from the baseline configuration and from the baseline performance.

3.1.2.1 Cell NT(II)-2-1 (1 mg/cm² Pt Anode)

Cell NT(II)-2-1 was fabricated with the major variations from baseline being a reduction in anode noble metal loading from 4 mg/cm² on the baseline configuration to 1 mg/cm². Previous attempts at reducing loading to this level using standard fabrication techniques resulted in cell performance well below normal. Those previous cells displayed visual pin point catalyst voids on the reduced anode catalyst. These voids were believed to have been caused by ambient moisture condensation on the anode during fabrication. In the fabrication of the anode for cell NT(II)-2-1 a technique was utilized that prevented condensation during fabrication. The resultant electrode was visually void free.

Cell NT(II)-2-1 was activated and performance tests revealed voltage levels nearly equivalent to baseline when considering the 10 mil (.254 mm) membrane vs. 5 mil (.127 mm) for the baseline configuration. The unplatinized 10 mil (.254 mm) membrane was utilized as a cost and schedule expedient. Figure 32 displays initial performance levels.

Cell NT(II)-2-1 was removed from the low pressure test facility and installed in the high pressure - high temperature facility. The cell was operated at 240°F and 115 psia pressure level. This temperature was selected as being near optimum for voltage performance at 115 psia from previous high temperature - high pressure testing. Figure 33 shows the DC voltage and Ir free voltage trend over three days of operation. As can be seen on Figure 33, the observed DC voltage decay was about equally related to internal resistance and non-resistance conditions.

Cell NT(II)-2-1 was disassembled for visual examination. When the cell exhibited a like new appearance, it was reinstalled into the test facility. Figure 34 indicates the continued performance decay during the second period of endurance testing. A second teardown revealed some membrane distortion in the hydrogen inlet area indicating subsaturated hydrogen fuel. This condition could well have caused chemical degradation, especially with unplatinized membrane, and resulted in
**TABLE II**

**PURPOSE:**

**TO DEMONSTRATE TECHNIQUES OF LOWERING CATALYST NOBLE METAL CONTENT WITHOUT LOSS OF OUTPUT PERFORMANCE.**

**TWO LIQUID ACID CELLS AND EIGHT SPE CELLS WERE FABRICATED AND EVALUATED AS PART OF THE TASK.**

<table>
<thead>
<tr>
<th>Fuel Cell Number</th>
<th>Deviation from 1975 Baseline Fuel Cell</th>
<th>Performance Variance from 1975 Baseline Fuel Cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>NASA (Phase II)</td>
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<td></td>
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<tr>
<td>Technology - Subtask - Cell</td>
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<td></td>
</tr>
<tr>
<td>NT(II) - X - X</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>O₂/H₂</strong> 10.0 ASF <strong>O₂</strong>/1.12 20.0 ASP VDC (0.761)</td>
</tr>
<tr>
<td><strong>NT(II)-2-1</strong></td>
<td>10 MIE SPE 28% H₂O₂</td>
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<tr>
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<td>Unplatinized Membrane,</td>
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<tr>
<td></td>
<td>1 mg/cm² Pt Anode</td>
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<tr>
<td><strong>NT(II)-2-2</strong></td>
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<td>+0.009</td>
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<td>Unplatinized Membrane,</td>
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<tr>
<td></td>
<td>1 mg/cm² Pt with 3 mg/cm² Graphite</td>
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</tr>
<tr>
<td></td>
<td>Anode (Layered)</td>
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<tr>
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<tr>
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<td>1 mg/cm² Pt Mixed with 3 mg/cm²</td>
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<td></td>
<td>Graphite Anode</td>
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<tr>
<td></td>
<td>3 mg/cm² Graphite</td>
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<td>Graphite (Layered)</td>
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<tr>
<td></td>
<td>Temperature 9nd Press.</td>
<td></td>
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<tr>
<td><strong>NT(II)-2-7</strong></td>
<td>.05 mg/cm² Pt and 3 mg/cm²</td>
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</tr>
<tr>
<td></td>
<td>Graphite on Cathode, high pressure</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Temperature 9nd Press.</td>
<td></td>
</tr>
<tr>
<td><strong>NT(II)-2-8</strong></td>
<td>.05 mg/cm² Pt Sputtered on</td>
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</tr>
<tr>
<td></td>
<td>4 mg/cm² Graphite Anode</td>
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</tr>
<tr>
<td></td>
<td>Gold Screen Cathode,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>HIGH PRESSURE FACILITY UNABLE TO OPERATE AT NEAR AMBIENT PRESSURES FOR COMPARISON.</td>
<td></td>
</tr>
</tbody>
</table>
Figure 32.

Fuel Cell NT(11)-2-1 (1 mg/cm² Pt Anode)

O - O₂/H₂ (L20°F)  R = 0.0105 Ohm
Figure 33.
Figure 34.

Fuel Cell NT(II)-2-1
115 Psia Oxygen
90% H₂O, O₂/H₂ ΔP
240°F

○ - DC Volts at 100 ASF
□ - IR Free Volts at 100 ASF

Days of Operation

Cell Voltage (Vols)
performance reductions. Corrective action for subsaturated reactants were not incorporated in the cells of this subtask until cell NT(II)-2-8, as the other cells were being evaluated in parallel with cell NT(II)-2-1.

In summary, cell NT(II)-2-1 showed that a reduction of anode noble metal loading to 25% of baseline could be incorporated with initial performance equivalent to baseline utilizing the fabrication technique that prevented anode catalyst voids. A major contributor to the performance decay with time is believed to be the subsaturated reactants.

3.1.2.2 Cell NT(II)-2-2 (1 mg/cm² Pt - 3 mg/cm² Graphite Layered Anode)

Cell NT(II)-2-2 was fabricated in a fashion identical to NT(II)-2-1 except a 3 mg/cm² layer of graphite Teflon mix was applied to the outside of the 1 mg/cm platinum anode loading. The purpose of the graphite layer was to increase electrical conductivity without interfering with the interface catalyst activity.

The cell was activated in the low pressure facility and showed performance levels slightly superior to the baseline configuration even with the 10 mil (.254 mm) membrane. (See Figure 35.)

The cell was then operated in a high temperature - high pressure facility. Figure 36 displays the 300 ASF (.323 ACM²) performance as the temperature was increased to 240°F. This figure shows that some performance decay was experienced during the overnight operation at 200°F. (Note that the increase humidification of reactants corrective action was not known at the time of operation of this cell.)

Endurance testing of cell NT(II)-2-2 at 240°F and 115 psia was continued for a total of seven days. (See Figure 37.) Performance decay did occur during this time, but at a much slower rate than cell NT(II)-2-1. Also, the performance loss was almost entirely related to non-resistance conditions.

The cell was disassembled for visual inspection and when a like new appearance was observed, it was reinstalled into the test fixture. Additional endurance testing was performed, also shown on Figure 37, which showed a continuation of the performance decay. During this second endurance period, product water analysis was performed during each working day. These analyses indicated a very high chemical degradation rate. (High HF release rates 15 to 30 times a normal cell under similar operating conditions.)

A second teardown of this cell revealed a rust colored material on the cathode wetproofing film. This material was identified as iron by chemical analysis. Iron is known to be a catalyst for hydrogen peroxide degradation of the electrolyte as well as a species that exchanges in the electrolyte thus reducing ion conductivity and
Fuel Cell NT(II)-2-2 (1 mg/cm² Pt - 3 mg/cm² Graphite Layered Anode

- O₂/H₂ (120°F) R = 0.009 Ohm

Figure 35.
Performance and Chemical Stability

Figure 37.
ultimately output performance. The source of this iron was found to be the 316 stainless steel reactant inlet lines. Corrective action of replacing these lines with non contaminating titanium was introduced on cell NT(II)-2-8.

In summary, cell NT(II)-2-2 displayed initial performance levels above baseline with only 25% of the amount of anode noble metal. The performance decay with time was probably greatly influenced by subsaturated reactants and iron contamination.

3.1.2.3 Cell NT(II)-2-3 (1 mg/cm² Pt - 3 mg/cm² Graphite Mixed Anode)

Cell NT(II)-2-3 was fabricated with the same amount and proportions of platinum and graphite on the anode except that they were thoroughly mixed instead of being in discrete layers. The purpose of this configuration was to confirm that the active interface is fixed at the outside surface of the membrane electrolyte and that only platinum at the interface contributes to the electrochemical reaction.

The operation of cell NT(II)-2-3, shown on Figure 38, did, in fact, reveal a performance level below that of cell NT(II)-2-2, thus tending to confirm the fixed interface postulation.

3.1.2.4 Cell NT(II)-2-4 (.02 mg/cm² Pt Sputtered Anode)

The sputtering technique of depositing a very low loading of platinum was first attempted on water electrolysis hardware with very encouraging results. The techniques were transferred to the fuel cell configuration on cell NT(II)-2-4. The anode configuration consisted of a pure graphite-Teflon electrode at the 3 mg/cm² loading level which was sputtered on the membrane side at the 0.02 mg/cm² platinum loading level.

Initial cell performance, shown in Figure 39, was significantly below baseline. However, the entire difference could be accounted for by the internal cell resistance. (Note the similarity in IR free performance between cell NT(II)-2-4 and baseline.)

The cell was operated in a high temperature - high pressure facility and demonstrated similar performance decay with time as was observed on cells NT(II)-2-1 and NT(II)-2-2. (See Figures 40 and 41.)

The major impact of this cell was the demonstration of the minute amount of platinum required in the anode electrode to perform its electrochemical function.

57
Fuel Cell NT(II)-2-3 (1 mg/cm² Pt - 3 mg/cm² Graphite Mixed Anode)

- O₂/H₂ (120°F) R = 0.0115 Ohm

Figure 38.
Figure 39.
Figure 40.
Figure 41.

Fuel Cell NT(II)-2-4
116 Palm Oxygen
96% H₂O₂ O₂/H₂ ΔP = 240°F

DC Volts @ 300 ASF

IR Free Volts @ 300 ASF
3.1.2.5 Cell NT(II)-2-5 (0.25 mg/cm² Pt Anode)

Cell NT(II)-2-5 was the second cell to utilize the layered anode catalyst approach. This cell was identical to cell NT(II)-2-2 except that the inner 1 mg/cm² layer was 75% graphite and 25% platinum instead of pure platinum. The purpose of this cell was to obtain additional information on the performance of cells vs. the amount of interface platinum and the amount of bulk platinum in the anode catalyst.

Figure 42 displays the initial performance of cell NT(II)-2-5 at 1200°F. This cell had virtually the same initial performance as cell NT(II)-2-3 which had four times the amount of bulk platinum but the same percentage of the platinum membrane interface contact. Once again, the IRfree performance was found to be nearly the same as the baseline configuration which contained 16 times as much bulk platinum.

Testing in a high temperature and pressure mode revealed similar performance and performance decay characteristics, as previous cells in this subtask. (See Figures 43 and 44.)

The major impact of this cell testing was a further confirmation of the minute amount of platinum required and that bulk platinum is unimportant relative to interface platinum when it comes to the pure electrochemical anode reactions.

3.1.2.6 Cell NT(II)-2-6 (.05 mg/cm² Pt Sputtered Cathode)

Cell NT(II)-2-6 was fabricated to demonstrate catalyst noble metal loading on the cathode electrode. A loading of 0.05 mg/cm² platinum was sputtered onto 3 mg/cm² graphite. It was found that the cathode electrode became detached from the membrane with normal pre-test handling. Since the detachment occurred in the middle of the graphite layer, leaving the sputtered portion attached, a second pure graphite layer at the 3 mg/cm² was attached to the first layer at higher than normal pressure and temperature. Although attachment was improved, an attempt at operation resulted in separation of the cathode from the membrane electrolyte.

3.1.2.7 Cell NT(II)-2-7 (.05 mg/cm² Pt Sputtered Cathode)

An attempt at fabricating a second cell with a platinum sputtered graphite cathode was made. This cell was fabricated with a single high temperature - high pressure press. Although this cell visually appeared firmly assembled, it also became separated upon attempted operation.

The experience obtained from cells NT(II)-2-6 and NT(II)-2-7 indicated that more assembly development of cathode electrodes would be required before
Figure 42.
Figure 43.
Figure 44.

Fuel Cell NT(II)-3-5 115 Ptulp Oxygen
99% H₂O O₂/H₂ ΔP
240°F

- DC Volts @ 300 ASF
- IR Free Volts @ 300 ASF
substantial cathode side noble metal reductions could be demonstrated.

3.1.2.8 Cell NT(II)-2-8 (.05 mg/cm² Pt Sputtered Anode)

Cell NT(II)-2-8 was fabricated with .05 mg/cm² platinum sputtered onto 4 mg/cm² graphite as the anode. The purpose of this cell test was to demonstrate the effect of introducing corrective actions into the test facility. The corrective actions that were incorporated were the change in the reactant inlet tubing material to titanium and increased reactant humidification.

Activation and operation in a high pressure - high temperature facility revealed equivalent to baseline initial performance at 100 ASF with a fall off below baseline at higher current densities. (See Figure 45.)

Figure 46 shows that a performance decay rate with time was even faster than had been experienced with the earlier cells. The HF release rate was, however, within the normal range. Some of the characteristics of the cell appeared very similar to cells that operated with contaminated fuel, (i.e., large performance improvement following high current density - low voltage operation). In this regard, fuel contamination is currently being investigated as a potential cause of the performance decay.

3.1.3 Materials Performance

It was the purpose of this subtask to investigate several materials/configurations that have the potential for increasing fuel cell performance and/or reducing overall fuel cell costs. A summary of the work performed in this subtask is as follows:

- A thin wetproofing film for cathode electrodes was found to have performance characteristics similar to the baseline configuration. Although a small cost reduction would result from introduction of this film, the main purpose of the investigation of demonstrating higher performance levels was not achieved.

- A cell containing a higher than normal IEC content was shown to exhibit higher electrical output performance. This amounted to 1 to 2% increase in cell efficiency.

- A low cost grooved anode collector plate was shown to have equivalent performance to baseline at low temperatures (120°F) but that the heat and mass balance was upset as temperatures were increased.
Figure 45.

Cell NT(II)-1-5 (1975 Baseline)
120°F - 115 Psia Cathode
\[ R = 0.0057 \]

Cell NT(II)-2-8
120°F - 115 Psia Cathode
\[ R = 0.0067 \]
Figure 46. Performance and Chemical Stability

Fuel Cell NT(II) 2-8
115 Psia Oxygen
110 Psia Hydrogen

- IR Free Volts @ 100 ASF
- DC Volts @ 100 ASF
- HF in Product H₂O

Days of Operation

Performance and Chemical Stability

Figure 46.
Carbon paper current collection previously demonstrated in water electrolysis technology, was attempted to solve the heat and mass upset caused by the grooved anode collector plate. Poor mechanical attachment revealed the need for additional development.

Initial low cost membrane performance was found equivalent to baseline performance. Refinement in assembly techniques were found to be necessary.

A total of five cells were fabricated and test evaluated as part of this subtask. Table III describes the variations from the baseline configuration and performance.

### 3.1.3.1 Cell NT(II)-3-1 (2 Mil Cheplast)

Fuel cell NT(II)-3-1, contained a 2 mil thick Cheplast wetproofing in place of the standard 7 mil thick material. Initial performance on both O_2/H_2 and air/H_2 was below the baseline performance (see Figure 47). After 11 days of operation and following several electrolysis activations, cell performance on air/H_2 had improved to slightly above baseline (i.e., 0.011 volt higher at 200 ASF).

The effect of the air humidifier temperature was then evaluated. With the fuel cell at 165°F, the air humidifier, which had been operating at 163°F for the duration of the test, was increased in temperature. Cell performance increased until the humidifier reached 165°F, and then voltage fall-off was noted (see Figure 48). A slow increase in voltage was noted when the heat input to the humidifier was removed and a slow cooling took place. When the humidifier temperature fall to about 153°F a rapid fall-off in voltage occurred. At 148°F the heat was reapplied to the humidifier and performance recovered as the temperature increased. At the end of the humidifier temperature evaluation run the cell was about 0.010 volt below the initial temperature evaluation performance. Overnight running, however, brought about full recovery of performance. Additional activation was performed, but no further performance increases were observed.

In summary, it can be stated that no significant advantage was demonstrated by the thin wetproofing.

### 3.1.3.2 Cell NT(II)-3-2 (High IEC Membrane)

Fuel cell NT(II)-3-2 was fabricated utilizing membrane with a .97 IEC vs. .8 IEC for the baseline configuration. The purpose of this cell test was to demonstrate improved output performance by use of a membrane with increased ion conductivity.
### Task 1.3 Materials Performance

**Purpose:**
To investigate several materials that have the potential for increasing fuel cell performance and/or reducing overall fuel cell costs.

Five fuel cells have been fabricated and evaluated as part of this task.

#### Performance Variance from 1975 Baseline Fuel Cell

<table>
<thead>
<tr>
<th>Fuel Cell Number</th>
<th>Deviation from 1975 Baseline</th>
<th>Performance Variance from 1975 Baseline Fuel Cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>NASA (Phase II)</td>
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<td>O₂/H₂ 100 ASF at 120°F</td>
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<tr>
<td>Technology - Subtask - Cell</td>
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<td>O₂/H₂ 200 ASF at 120°F</td>
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<td>NT(II) - X - X</td>
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<td>O₂/H₂ 100 ASF at 120°F</td>
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<td>O₂/H₂ 200 ASF at 120°F</td>
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<td></td>
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<td>Air/H₂ 100 ASF at 120°F</td>
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<td>Air/H₂ (10 PPM CO) 100 ASF at 120°F</td>
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<td>Air/H₂ (10 PPM CO) 200 ASF at 120°F</td>
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</table>

16 PIAA Cathode
15 PIAA Anode
Fuel Cell NT-6-2 (1975 Baseline)

- O$_2$/H$_2$ (120°F) $R = 0.0067$ Ohm
- O$_2$/H$_2$ (165°F) $R = 0.0066$ Ohm
- Air/H$_2$ (165°F) $R = 0.007$ Ohm
  2.5 x Stoich
- O$_2$/H$_2$ (25% CO$_2$ - 0.3% CO)
  1.25 x Stoich (165°F) $R = 0.0065$ Ohm
- O$_2$/H$_2$ (25% CO$_2$ - 0.3% CO)
  1.25 x Stoich (165°F) $R = 0.0065$ Ohm

Fuel Cell NT(II)-3-1 (2 Mil Chemplast)

- O$_2$/H$_2$ (120°F) $R = 0.0075$ Ohm
- O$_2$/H$_2$ (165°F) $R = 0.0062$ Ohm
- Air/H$_2$ (165°F) $R = 0.0062$ Ohm
  2.5 x Stoich Air (Initial Performance)
- Air/H$_2$ (185°F) $R = 0.006$ Ohm
  2.5 x Stoich Air (11 Days After Start)

Figure 47.
Fuel Cell NT(II)-3-1
Performance @ 165°F
16 Psia Air
15 Psia H₂

Figure 48.
A low pressure performance check showed an improvement over the baseline cell of approximately 0.02 volt at 200 ASF. The cell was then installed in a high temperature - high pressure facility and was operated on pure hydrogen and oxygen from 15 to 115 psia and from 75°F to 300°F. Figure 49 displays the performance of cell NT(II)-3-2 at 115 psia, 20 inches H₂O, ΔP and 300°F. Unlike cell NT(II)-1-2, this cell did not have a clearly defined optimum performance below 300°F. Figure 50 shows the cell performance of cell NT(II)-3-2 versus temperature at 100 and 300 ASF. Although the highest voltage at 100 ASF was obtained at about 260°F, the 300 ASF performance was about the same at 280 and 300°F.

In summary, cell NT(II)-3-2 demonstrated two characteristics of a positive nature:

- Higher than baseline performance.
- Higher than baseline optimum performance temperature.

3.1.3.3 Cell NT(II)-3-3 (Grooved H₂ Collector Plate)

Cell NT(II)-3-3 was fabricated with a low cost grooved H₂ collector plate configuration. The initial O₂/H₂ performance (shown on Figure 51) was about equivalent to baseline at 120°F when corrected for the difference in resistance. At 165°F, however, the cell appeared to be current limited at slightly above 200 ASF.

Cell NT(II)-3-3 was operated for several days on O₂/H₂ during which time the performance remained stable. The oxidant was switched from pure oxygen to air with a flow rate 2.5 times the stoichiometric requirements. Very unstable performance was quickly evidenced (i.e., @ 100 ASF cell voltage cycled from .68 volt to .52 volt every 3 to 5 minutes). Increased hydrogen side purging had no effect on performance. The load was reduced to 50 ASF at 5 times required air flow and still the same type of cycling continued.

The cell was operated for two days on air at both 50 and 100 ASF and the cycling continued throughout. Internal cell resistance was observed to be slightly reduced from original at the 0.0075 ohm level. A switch back to pure oxygen was made and the cycling stopped. Performance at 100 ASF was approximately .080 volt below the pre-air performance. The cell was operated for four additional days during which time the performance continued to decay and the cell became current limited to about 80 ASF. Resistance remained constant throughout this period.

Cell NT(II)-3-3 was removed from test and disassembled for inspection. The only visual variant condition observed was an anode catalyst striplike "plating" which was coincident with the collector plate grooves. The cell was reassembled utilizing the baseline gold screen anode current collector configuration. A repeat test on O₂/H₂ showed similar performance and resistance as just prior to the first test termination (i.e., current limited to 80 ASF).
Fuel Cell NT-12-3 (High IEC)

- O₂/H₂, (250°F), 100 psig O₂, 20" H₂O ΔP
- R = 0.0049 Ohm

Figure 49.
Figure 50.

Cell Volts - DC @ 100 psig O₂ and 15³ H₂O ΔP

R = 0.0060

R = 0.0054

R = 0.0052

R = 0.0051

Fuel Cell N°(11)-3-2 Performance

Fuel Cell Coolant Temperature - °F

Figure 60.
Figure 51.

**Fuel Cell NT-6-2 (1975 Baseline)**

\[\bigdiamond\ - \ O_2/H_2 \ (120^\circ F) \ R = 0.0067 \text{ Ohm}\]

\[\bigtriangleup\ - \ O_2/H_2 \ (165^\circ F) \ R = 0.0065 \text{ Ohm}\]

**Fuel Cell NT(II)-3-3 (Grooved H}_2\text{ Collector)**

\[\bigcirc\ - \ O_2/H_2 \ (120^\circ F) \ R = 0.0098 \text{ Ohm}\]

\[\square\ - \ O_2/H_2 \ (165^\circ F) \ R = 0.0085 \text{ Ohm}\]

Grooved H}_2\text{ Collector Performance
Fuel cell NT(II)-3-3 was removed from the test fixture and exchanged in H$_2$SO$_4$. This process was accomplished to rule out contamination of the solid polymer electrolyte. Following exchange, the cell was reassembled into the test fixture, reactivated and operated. No change in performance was noted and resistance remained in the normal range.

Fuel cell NT(II)-3-3 was removed from the test fixture and sent for chemical analysis. The only variant condition detected was that the water content of the SPE had reduced from a normal 37% to 20%.

The conclusion reached was that the configuration upset the heat and mass balance to the point of producing a drying mode of operation even though reactants were suitably pre-humidified.

3.1.3.4 **Cell NT(II)-3-4 (Carbon Paper Collector)**

Fuel cell NT(II)-3-4 was fabricated with a non wetproofed carbon paper attached to the anode catalyst. This was done to reduce the anode side IR heating in conjunction with the grooved collector plate and thus correct the heat and mass balance. Activation and operation was unsuccessful as the carbon paper became detached from the anode catalyst before useful data could be taken.

3.1.3.5 **Cell NT(II)-3-5 (R.A.I. Membrane)**

The low cost RAI membrane was fabricated into cell NT(II)-3-5. This cell differed from the baseline in that the unplatinized RAI membrane was utilized and the cathode contained a gold current collector screen. The low pressure performance as shown on Table III was found about equivalent to baseline performance. The high pressure performance, as compared to cell NT(II)-1-4, is displayed on Figure 52. The slightly lower performance of cell NT(II)-3-5 can be accounted for by the somewhat higher internal resistance, (i.e., the RAI membrane was 7 mils thick vs. 5 mils for the baseline membrane).

Three observations began to form trends as testing continued:

- HF release in the product water was considerably higher than that experienced with Nafion® under similar operating conditions.
- Internal cell resistance increased with time, (i.e., 50% increase over two weeks of operation).
- Reduced cell output performance commensurate with the increase cell resistance.
Figure 52.

- Cell NT(II)-3-5 165°F - 115 psia cathode
  $90^\circ$ H$_2$O - O$_2$/H$_2$ $\Delta P$
  $R = 0.0059$

- Cell NT(II)-1-4 165°F - 115 psia cathode
  $90^\circ$ H$_2$O - O$_2$/H$_2$ $\Delta P$
  $R = 0.038$

RAI Membrane Performance

Current - ASF
Cell NT(II)-3-5 was disassembled for visual examination with anode catalyst detachment of approximately 15% noted.

Since the cell did not contain the membrane stabilization catalyst, the higher HF release rate might have been expected. The higher resistance/lower performance appeared at least partly the result of the detached anode catalyst.

Platinization trials were performed on RAI membrane samples with results being found superior to the Nafion ® film, (i.e., complete platinization throughout the thickness of membrane). Attachment procedures will now have to be modified to assure adequate electrode attachment.

3.2 Stack Test Evaluation

The purpose of this task was to conduct a thorough performance analysis on the single 0.7 Ft² (650 cm²) cell stack NT-01 produced during Phase I. The major purpose of fabricating and testing this large sized cell was to demonstrate the scale up capability of the configurations being analyzed in the 3 x 3 hardware, (i.e., this cell size was approximately twice the size of any previous space type solid polymer electrolyte cell).

The initial activation and operation of the large scale cell stack (NT-01) was conducted without difficulty. However, as the operational temperatures were increased toward 120°F, an ever increasing fuel side restriction was observed. Since the restriction prevented the introduction of fuel at the desired rate, the current density was severely limited.

A unit teardown was conducted to determine the source of the fuel side restriction. Figure 53 displays the coolant cartridge assembly which forms the fuel chamber between it and the membrane electrode assembly. Note the frame extrusion into the hydrogen manifold slot. This extrusion was identified as the cause of the restriction.

Reassembly of the unit was performed utilizing a fiberglass reinforced frame.

Reactivation of the unit showed that the restriction had been eliminated. Operation continued for over 300 hours when an automatic shutdown occurred due to an open circuit within the stack. A second teardown revealed a badly oxidized terminal current collector plate. (See Figure 54.) This aluminum terminal plate oxidized when moisture from the cathode system found its way into this area of the stack.

The terminal plates were replaced with niobium material and one damaged coolant cartridge was replaced. Shortly after reactivation, a facility
Figure 53. Hydrogen Chamber

Source of $\text{H}_2$ Restriction
ventilation failure resulted in stack automatic shutdown. Membrane damage was sustained during the shutdown probably due to a hydrogen/oxygen gas mixture trapped in the instrumentation as a result of the normal oxygen takeover.

The membrane-electrode assembly was replaced, because to repair the effected area would have significantly reduced the active area. The stack was re-identified as NT-02 to reflect the change in membrane electrode assembly. The operating procedure was modified so as to prevent cell damage during automatic shutdown.

Reactivation was performed and full operational temperature conditions were reached. Figure 55 displays the resultant performance both on O₂/H₂ and air/H₂. Stack NT-02 has been operated for short periods on highly contaminated fuel, (i.e., 74% H₂, 24.6% CO₂, .3% CO and .1% CH₄) with expected performance results. The stack NT-02 has currently exceeded 1600 hours of operation with invariant cell performances. Continued endurance testing is planned for this unit.

As additional hours are accumulated on stack NT-02 the demonstration of scale up capability is further enhanced.
Fuel Cell Stack NT-2
Cell Area - 0.7 Ft$^2$
Cathode Pressure - 16 Psia
Anode Pressure - 15 Psia

- $O_2/H_2$ at 120°F
- $O_2/H_2$ at 165°F
- AIR/H$_2$ at 165°F

Large Scale Cell Performance
Figure 55.
4.0 CONCLUSIONS

The conclusions reached during this Phase 2/2A technology program can be summarized by categorizing the various areas of investigation into one of the following groups:

- **Demonstrated advances** - items which resulted in an improvement without any detectable detrimental effects.
- **Promising areas** - items which showed some limited success with the promise of further advances.
- **Potential areas** - items which were unsuccessfully demonstrated but are still considered candidates for further advances.
- **Low probability areas** - items which were unsuccessfully demonstrated and which are considered unlikely to be advanced in the near term.

4.1 Demonstrated Advances

4.1.1 Temperature Pressure Performance

Very significant improvements in cell performance levels and current densities were demonstrated at increased temperature and pressure conditions, (i.e., ≈ 9% efficiency increase at 500 ASF and a three-fold increase in maximum stable current density from 500 to 1500 ASF). With identified corrective actions of increased reactant humidification and reactant inlet lines material changed from 316 stainless steel to titanium, the increase performance and current density was established without apparent loss of long-term performance and chemical stability.

4.1.2 High IEC Membrane Performance

The testing of the high IEC membrane cell resulted in a performance improvement of 1 to 2 percent on efficiency. Although this may not seem significant at first inspection, this could be very significant for long term missions with its considerable reactant weight reductions due to this improved cell efficiency.

The limited amount of testing performed on this hardware showed no evidence of performance or chemical degradation with operational life.
4.1.3 Scale Up Capability

The question of ability to scale up a fuel cell design from small laboratory hardware to full scale hardware was demonstrated in the fabrication and test evaluation of single cell stacks NT-01 and NT-02. The anomalies uncovered during the fabrication and testing were quite easily solved with a resultant 1600 hours of demonstrated invariant performance on a cell twice the active area of the largest previously fabricated space type fuel cell.

4.2 Promising Areas

4.2.1 Catalyst Performance

The catalyst noble metal reduction task demonstrated several methods of obtaining equivalent performance with significant noble metal reductions in the anode electrode. However, performance stability with time was not achieved. Additional efforts will be required to achieve this performance stability. Success in this activity will result in considerable cost reduction in electrode raw materials.

4.2.2 Low Cost Membrane Performance

The low cost membrane task resulted in equivalent to baseline cell performance. The impact of introduction of low cost membrane would be a 50 to 75% reduction in the basic cost of the solid polymer electrolyte. Refinement of assembly techniques utilizing the low cost material are still required to obtain the desired electrode to membrane attachment.

4.3 Promising Areas for Advancement

4.3.1 H₂ Distribution Performance

A new anode side gas distribution and current collector configuration was attempted but would have to be generally categorized as being unsuccessful. The simplified low cost configuration appeared to upset the heat and mass balance of the operating cell, (i.e., low temperature was equivalent to baseline but performance deteriorated with increasing temperature). The means of improving the heat and mass balance of this revised configuration is thought to be readily available but additional development is required.

4.4 Low Probability Areas

4.4.1 Thin Wetproofing Film Performance

The test conditions that were thought to require a lower wetproofing pressure drop were those where the oxygen partial pressure was very low. The thin
wetproofing film had a considerably reduced pressure drop. The thin wetproofing cell, when operated at very low oxygen partial pressures (2-3 psi), showed performance levels only equivalent to the thicker wetproofing cell. It appears that improved performance with the thin wetproofing material is unlikely.
5.0 RECOMMENDATIONS

The recommendations for the near term technology advancement activities include continued laboratory testing of 3 x 3 cell and a system study to assess the potential impact of the technical advancements made during Phase I/1A and Phase 2/2A. Figure 56 displays the schedule for the recommended NASA/JSC Technology Program – Phase 3. Details of the recommended program are as follows:

5.1 Task 1.0 – Continued Laboratory Evaluations:

This task is divided into two main subtasks both of which will be directed toward the demonstration of a cost effective technology in laboratory 3 x 3 hardware.

5.1.1 Subtask 1.1 – Configurations Analysis:

This subtask will consist of short term 3 x 3 cell investigations of operational parameters and configuration modifications.

Item A – High Current Density Evaluations:

During the past year current density increases of over three-fold were demonstrated (i.e., 500 ASF to over 1500 ASF). Cell stability and max current density appeared to be limited by excess moisture content in the hydrogen gas stream. Laboratory hardware is currently being developed to internally trap excess anode side moisture and thus allow a true assessment of high current density stability and limiting current. One or more cells will be fabricated and evaluated in this new hardware configuration. This hardware will also have all the characteristics required for high current density, high temperature and high pressure.

Item B – High Operational Temperature Evaluations:

Considerable operational data was accumulated at high cell temperatures during the past year. However, some performance instability was noted as operational temperatures approached 300°F. Excess anode side moisture is also believed to be the cause of this condition. The new internal anode trap hardware will be utilized to evaluate one or more cell configurations at the higher cell temperatures.

Item C – Catalyst Evaluations

As many as five cell configurations showed good promise for significant catalyst noble metal loading reductions during the past year's effort. To date none of
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**NASA/JSC TECHNOLOGY PRELIMINARY PROGRAM PHASE III**

Figure 56.
these configurations have demonstrated the invariant performance typical of the baseline configuration. As part of this work element investigations will continue to determine the cause of the non-invariant performance. In addition, as developments are made through the water electrolysis technology and IR and D efforts new noble metal reduction techniques will be evaluated in the 3 x 3 hardware.

Item D - Electrolyte Evaluations

A new membrane polymer (RAI grafted FEP) showed performance levels nearly equivalent to Nafion with projected costs of the RAI material of less than 50% of the Nafion. Two undesirable characteristics have been encountered to date:

- HF releases in the product water is higher than with Nafion.
- Performance decay with time was observed.

The RAI membrane that was tested did not contain the platinization process which was added to the Nafion to increase chemical stability. Tests are in process to determine the optimum platinization procedure for RAI membrane. This procedure should be determined prior to the 1977 effort.

The performance decay was found to be associated with detachment of the anode electrode. The attachment procedures for platinized RAI membrane will be investigated during the 1977 program and will be incorporated into one or more cells and performance evaluated.

5.1.2 Subtask 1.2 - Endurance Evaluations:

A series of endurance test facilities will be set up and test evaluations conducted. Initially the tests will include:

- Intermediate Temp. - Intermediate Current Density
- Intermediate Temp. - High Current Density
- High Temp. - Intermediate Current Density
- High Temp. - High Current Density

These initial endurance cells will be of a configuration similar to the 1976 baseline configuration. As configurations from Subtask 1.1 reveal suitable promise, endurance tests will be started on these.
5.2 Task 2.0 - System Study:

The SPE technology advancements over the past two years have been rigorously evaluated from a system viewpoint. General impacts have been estimated in terms of dollars per KW, weight per KW and the like, but a specific system study incorporating these advancements has not been conducted.

With input specifications from NASA/JSC a system study will be performed to determine the system characteristics. Items to be determined will include:

- Heat rejection requirements
- Specific reactant consumption
- Parasitic power requirements
- System weight analysis
- Cost analysis

5.3 Planned IR&D Supporting Activities

In addition to the NASA/JSC funded program GE/DECP intends to support fuel cells in the IR and D program. In this regard the use of NASA property (fuel cell stack NT-02) is hereby requested. Additional life endurance characteristics of this hardware, and perhaps modifications thereof, will be conducted under IR and D funding. Results of such testing would be reported in the monthly technical reports to NASA/JSC.

Significant progress in cathode conductive wetproofing and its related simplified bipolar construction has been made during the 1976 IR and D program. Continued efforts in this area are planned in the 1977 IR and D program. This effort may include scale up cell hardware suitable for space oriented fuel cells.

Continued endurance testing of long lived units under IR and D funding also are planned for continued fuel cell support. These units include:

- AFC #6 O₂/H₂ - 180°F - 70 psia > 39,000 hours
- PP #1 Air/H₂ - 80°F - 14.7 psia > 16,000 hours
- PP #3 Air/H₂ - 80°F - 14.7 psia > 9,000 hours