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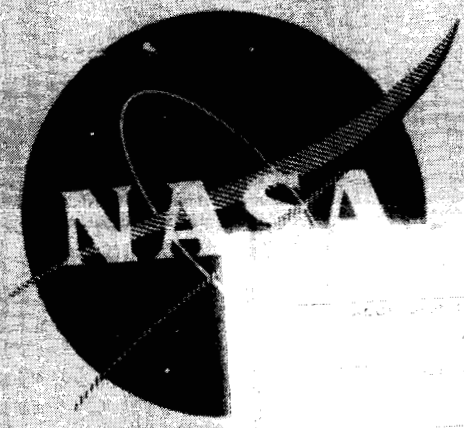

Development of High-Performance Light-Weight Electrodes for Hydrogen-Oxygen Fuel Cells

FIRST QUARTERLY REPORT

April 5, 1965 – July 5, 1965

by

D. Gershberg, Principal Investigator

W. P. Colman

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NATIONAL AERONAUTICS AND SPACE ADMINSTRATION

CONTRACT NAS 3-6477

AMERICAN CYANAMID COMPANY

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November 15, 1965

CONTRACT NAS 3-6477

Period Covered: April 5, 1965 - July 5, 1965

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1. INTRODUCTION

1.1 Objectives

Light-weight fuel cell batteries capable of producing large quantities of energy appear feasible for space applications. High-performance light-weight electrode systems are an essential part of these batteries. Work completed previously[1], under NASA Contract NAS 3-2786, showed that American Cyanamid AB-40 electrodes (40 mg Pt/cm^2) give high and sustained performance in hydrogen-oxygen matrix fuel cells including those of battery size; this performance is substantially higher than that of American Cyanamid AB-1 electrodes which contain less platinum (9 mg Pt/cm^2). It was calculated that at temperatures up to 100°C, the AB-40 electrodes could be incorporated in a 2 kw fuel battery whose weight per net power (including all auxiliaries except fuel and tankage) would be approximately 50 lb/kw.

A detailed investigation at temperatures up to 100°C showed that initial performance generally increases with increasing temperature, pressure, and electrolyte (KOH) concentration. Furthermore, preliminary studies demonstrated that substantial increases in initial performance can be obtained by operating at higher temperatures (140°C) and KOH concentrations (65%), than are generally employed in matrix fuel cells. Under these conditions current densities as high as 100, 400 and 800 ma/cm^2 at working voltages of 1.0, 0.9 and 0.8 v, respectively, were achieved in short term tests.

Accordingly the objective of the present contract is to investigate and recommend preferred conditions, at 100-200°, under which AB-40 electrodes would be capable of sustained high performance in a total module having a weight-to-power ratio substantially lower than those presently available for space environment.

1.2 Scope

The scope of work to be done by American Cyanamid Company is given by Article I-B in the Statement of Work of Contract NAS 3-6477.

Article I-B Specific Work Requirements

The contracted effort shall be performed in accordance with the following outline to the extent necessary to meet the objective of each task.

1. Task I - Small Cell Testing

   I-A. Short-Term Testing

   a. Investigation of Operating Variables

      The effect of assembly and operating variables on initial cell performance and on internal cell resistance shall be determined in cells having an active area of 5 square centimeters. The following variables shall be investigated over the ranges indicated:

      Current Density: 100 - 1000 ma/cm^2
      Temperature: 100 - 200°C
      Pressure: 0 - 60 psig
      Electrolyte (KOH) Concentration: 30% by weight to the solubility limit.
      Electrolyte Loading: Variable, depending on matrix.
      Reactant Moisture Content: 0 - 90% saturated with respect to cell conditions.
      Fraction of Product Water Removed at each electrode: 0 - 1

      The actual terminal voltage shall not be less than 0.65 volts.

      Data to be obtained in Task I-A-a shall consist of polarization curves and internal resistance measurements.

   b. Investigation of Materials

      Stability of matrix, current collector screens, and gasket materials shall be determined. The cell component materials investigated shall include at least the following:

      (1) Matrices

         Fuel Cell Asbestos
         American Cyanamid ACCO Asbestos
         Polypropylene Felt
         Porous PTFE

         Measurements shall be made of the pressure differential (bubble pressure) required for massive transport of gas through the matrix materials used in this investigation.

(2) Current Collector Screens

Nickel Screens plated with either gold or rhodium.

(3) Gaskets

Armalon
Silicone Rubber-Encased in Teflon Tape
Porous PTFE
Solid PTFE

The objective of Task I-A-b is to obtain stable performance in cells having an active area of 5 square centimeters for a period of 70 hours defined as a cell voltage decline of not more than thirty (30) millivolts. The investigations shall be carried out over the range of operating variables listed in Task I-A-a. Initial investigations shall be conducted in the temperature range of 140°-175°C.

The contractor shall recommend the operating conditions and cell components suitable for long term operation for review and approval by the cognizant NASA Project Manager.

Should the matrix selected for life testing be different than the one used for the study of operating variables, Task I-A-a, small cell tests shall be run with this matrix under preferred conditions and a direct comparison of the results obtained in all phases of the program made.

c. Start-Up and Shut-Down Procedures

The practicability of start-up and shut-down procedures shall be investigated for cells operating at electrolyte concentrations greater than 50% potassium hydroxide by weight.

(1) For start-up the cells shall be loaded with a solution of potassium hydroxide, 50% by weight, and then concentrated to the desired level by excess gas flow while the cell is in operation.

(2) The shut-down procedure shall attempt to dilute the electrolyte concentration to 50% or less by either reduction of gas flow, or increasing the reactant moisture content while lowering the cell temperature.

I-b. Long-Term Testing

Small scale life testing shall be conducted in two inch by two inch active area cells of the same design as employed in Contract NAS 3-2786.

Life testing shall begin immediately. Preferable assembly and operating conditions shall be employed when defined in Task I-A and approved by the cognizant NASA Project Manager.

Fifteen cells shall be operated at these conditions under constant current loads in the range of current densities between 100 and 1000 milliamperes per square centimeter with the objective of obtaining stable performance. Stable performance is defined as a cell voltage decline of not more than 50 millivolts in a period of 1200 hours. Cell resistance shall be measured throughout each run.

2. Task II - Large Cell Fabrication

The contractor shall fabricate three (3) cells, 6 inch by 6 inch active area, for laboratory single cell polarization and life testing. A list of materials and design specifications shall be submitted to the cognizant NASA Project Manager for approval prior to fabrication.

3. Task III - Large Cell Testing

Testing in the 6 inch by 6 inch battery size cells shall be started at once. Preferred assembly and operating conditions shall be employed as soon as defined in Task I-A and approved by the cognizant NASA Project Manager. The cell components shall be those suitable for long term operation. This work shall have the objective of verification of conditions of preferred initial performance and demonstration of life performance. In addition, attempts to measure electrolyte concentration gradients shall be made.

a. Verification of Optimum Initial Performance

Using the previously defined operating conditions, performance in 6 inch by 6 inch cells shall be determined at current densities of 100-1000 milliamperes per square centimeter and compared with that obtained in 1 inch cells in Task I-A-b.

Data to be obtained in Task III-a shall consist of polarization curves and internal resistance measurements.

b. Long-Term Operation

Four 6 inch by 6 inch cells shall be operated under constant current loads at current densities of 100-400 milliamperes per square centimeter with an initial working voltage of not less than 0.85 volts. The objective is to obtain stable performance, defined as a cell voltage decline of not more than 60 millivolts in a period of 1,000 hours.

c. Effect of Electrolyte Concentration Gradients on Cell Performance

Electrolyte concentration gradients shall be measured over the face of the matrix at the termination of selected long term tests.

d. Start-Up and Shut-Down Procedures

The feasibility of start-up and shut-down by the methods developed in Task I-A-c shall be investigated during short term runs of up to 100 hours.

4. Task IV - Experimental Data Evaluation

The experimental data shall be analyzed and operating conditions shall be recommended in terms of the following cell parameters:

a. Component Specifications
b. Reactant pressures and moisture content
c. Operating temperature
d. Electrolyte concentrations and loading
e. Fraction of total water removal at each electrode
f. Method for cell start-up and shut-down

2. SUMMARY

During the first quarter of the contract, work was performed on Tasks I-A and I-B. Since a suitable matrix is essential to carry out all of the tasks, the effort on Task I-A was directed primarily toward evaluating potential matrix materials.

Task I-A

1. A literature search was made of the major abstracts of unclassified government contract work during the past 3-4 years. The search revealed no newly developed non-metallic material to be available in a form which might be used as a matrix for KOH at temperatures up to 200°C.

2. Eleven available and promising matrix materials, both commercial and proprietary, were evaluated. These included one or more forms of asbestos, PTFE*-Asbestos, porous PTFE and zirconia. It was found that none of these matrices possesses all of the properties (resistivity, corrosion resistance, mechanical strength and bubble pressure) to the degree required for the investigation of operating variables at temperatures up to 200°C and pressures above atmospheric. However, some of these matrices were found to be useful over more limited ranges of temperature and pressure.

a. Asbestos matrices are suitable for obtaining initial performance data at temperatures up to 150°C. The lowest resistivity and highest initial performance are given by proprietary ACCO-I Asbestos, ACCO-II Asbestos and commercial Fuel Cell Asbestos in that order. ACCO-I Asbestos is limited to atmospheric pressure operation. ACCO-II Asbestos, as well as Fuel Cell Asbestos can be used at pressures above atmospheric.

*polytetrafluoroethylene

b. The proprietary PTFE-Asbestos matrix gave approximately the same resistivity and initial performance as Fuel Cell Asbestos and permitted initial performance data to be obtained at temperatures up to 200°C. It is useful only at atmospheric pressure.

c. Commercial zirconia paper has the lowest resistivity of any of the matrices evaluated and gave the highest initial performance at current densities of 300-1000 ma/cm^2. The paper permitted initial performance data to be obtained at temperatures up to 200°. (For long term performance in KOH zirconia is limited to lower temperatures.) However, zirconia paper was found to be unsuitable for the investigation of operating variables because its use is limited to atmospheric pressure and because its performance was not always reproducible.

d. The outstanding corrosion resistance of PTFE makes it potentially suitable as a matrix material for long term as well as short term operation. The possibility of using PTFE as a matrix despite its extreme non-wettability in fuel cell electrolytes was demonstrated both with Teflon felt and microporous PTFE sheet. This was accomplished by adding perfluorinated surfactants (themselves stable in KOH up to 150-200°C) to the KOH in amounts which did not cause simultaneous flooding of the electrodes. By this means a commercial microporous sheet gave the same initial performance as Fuel Cell Asbestos while a proprietary matrix gave higher performance. The initial performance of commercial PTFE felt was poorer. The negligible bubble pressure of these matrices (< 4 in. water) limits their usefulness to atmospheric pressure only.

The various types of microporous PTFE sheets were found to be generally unsuitable at the higher end of the temperature range because they flowed under compression, allowing the electrodes to make contact and short circuit the cell.

3. The matrices available at present make it possible to investigate the effects of operating variables either at 100-200°C at atmospheric pressure only or at 100-150°C over the entire pressure range of 0-60 psig. The latter option was chosen mainly because it does not require the elimination of an important variable. A statistical design for the investigation of operating variables originally devised for the temperature range 100-200°C was revised for the range 100-150°C. The design permits a mapping of temperature, pressure, and KOH concentration effects. At preferred conditions of these three variables, the effects of reactant moisture content and the fraction of water removed at each electrode is to be investigated. ACCO-II asbestos matrix will be used in this work.

4. During the course of the materials investigation, very high performance was achieved at 200°C and atmospheric pressure, with KOH concentrations of 75-80%. Working voltages of 1.07, 0.96 and 0.76 were obtained at current densities of 100, 400 and 1000 ma/cm^2 respectively. This further confirms the conclusion reported previously[1] that cell performance generally increases with increasing temperature and KOH concentration. It also demonstrates the potential performance advantage to be gained by operating a matrix fuel cell at temperatures as high as 200°C (approximately 50 mv and 100 mv compared to the performance at 150°C and 100°C) provided that a suitable matrix is developed.

Task I-B

1. In small cell life testing at 100°C, ACCO-I asbestos matrices gave both a higher initial voltage and better voltage stability than Fuel Cell Asbestos. Voltage stability was improved by pre-wetting the electrodes with KOH before assembling them in the cell. At current densities of 100, 200, and 300 ma/cm^2, the lowest voltage decline rates (0.6, 3.1 and 4.2 mv/100 hrs.), during 500, 800, and 1050 hours respectively, did not exceed the maximum decline rate desired for a 1200 hour period (4.2 mv/100 hrs.). Final working voltages at these current densities were 0.95, 0.90 and 0.85 respectively.

2. With ACCO-I asbestos at 125°C, voltage stability, thus far, has been poorer than at 100°C despite improvements in stability obtained by pre-wetting the electrodes. Preliminary evidence indicates that the voltage decline at 125°C may be at least partly attributable to blockage of the electrode pores resulting from corrosion of the matrix.

3. SMALL CELL TESTING

3.1 Test Cell Design

Small scale fuel cells are being used in two sizes. Cells having a one-inch circle active area (5 cm^2) are used for obtaining polarization data. For life testing, cells having a 2" x 2" active area (25.8 cm^2) are of two types, one for use at atmospheric pressure and another for pressures up to 60 psig. The cells and their components are the same as those described previously[1] except for the following modifications.

1. To prevent corrosion by KOH at temperatures up to 200°C, all electrode screens and spacer screens are coated with 0.05 mils of gold. Cell face plates are triple-coated with 0.5 mil gold, 0.1 mil rhodium, and 0.5 mil gold.

2. In one-inch cells, the edges of the matrix are encased in heat-sealed Aclar polychlorotrifluoroethylene film (Allied Chemical) to prevent evaporation of water from the electrolyte. The Aclar film withstands higher temperature than the polyethylene film used previously.

3. In two-inch atmospheric pressure cells, the periphery of the matrix was previously sealed by compression between two thin sheets of PTFE (having a square opening in the center) which themselves were sealed between PTFE gaskets. The present design, shown in Figure 3-1, replaces the two thin PTFE sheets by a single thin PTFE sheet which frames the matrix but does not compress it. The matrix is compressed directly by the gaskets to the same thickness as the frame.

3.2 System Design and Operation

The equipment used for the determination of polarization curves and for life testing, exclusive of the cells themselves, was described previously[2]. The hydrogen is a pre-purified grade (Airco) nominally containing approximately 0.1 ppm O_2, 1 ppm CO and 3 ppm CO_2. The oxygen (Airco) contains approximately 5 ppm CO_2.

2" x 2" CELL: ATMOSPHERIC PRESSURE DESIGN

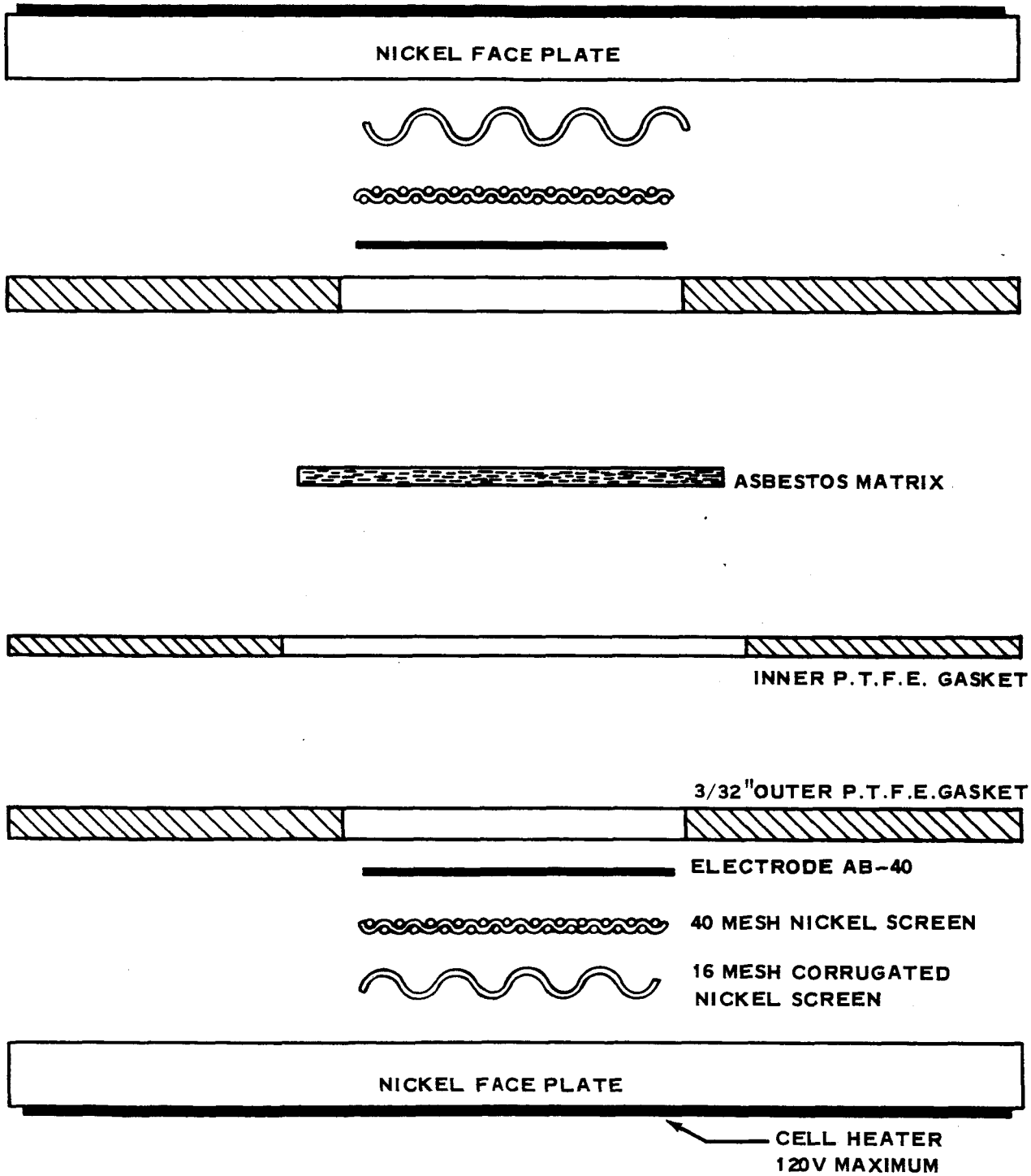

FIGURE 3-1

3.2.1 Polarization Curve Procedure

In starting a run, the matrix is filled with a pre-determined amount of electrolyte and then assembled in the cell. The cell bolts are tightened with a torque wrench to a pressure of 150-180 psi. The cell is heated to the desired temperature, during 5-15 minutes, and then is controlled to within $\pm$ 1°C. The gases are admitted to the cell at the rate of 2-4 cc/min. When the maximum open circuit voltage is reached, usually within 5 minutes, polarization measurements are made at increasing current densities up to 1000 ma/cm^2, generally in increments of 100-200 ma/cm^2. At each current density, the electrolyte concentration is maintained constant by setting the gas flow rates to remove product water as fast as it forms. All voltages reported are working voltages. At each current density, the voltage is considered initially stable if it remains constant to within 3 mv for 1-2 minutes.

After reaching 1000 ma/cm^2, a second polarization curve is run, again at increasing current density. It was reported previously[1] that for AB-40 electrodes the second polarization curve voltages are generally 20-40 mv above those of the first curve at current densities up to 500-600 ma/cm^2, but this difference tends to disappear at still higher current densities. Since no further changes occur during third polarization curves the performance data reported are those of the second polarization curves.

The room temperature solubility of KOH in water is 52%. For runs at higher concentrations, 50% KOH was loaded into the matrix and then was concentrated to the desired level during the first polarization curve determination by setting the gas flow rates to maintain that level.

3.2.2 Life Testing Procedure

Life tests are run on dry gases or on gases which are partially saturated with water vapor (with respect to cell humidity) before they enter the cell.

For dry gas operation the start-up procedure is the same as that described in section 3.2.1 for obtaining polarization data except that the run is started immediately at the current density desired for the test. The cells are run at constant current. Voltage and temperature are recorded continuously, and internal resistance is measured daily.

Stable cell operation requires maintaining the amount of water in the cell constant within certain limits by removing water from the cell at the same rate as it is formed by the cell reaction. Performance losses can result from changes in the average or local concentration of electrolyte, or from changes in the quantity of electrolyte solution held in the matrix and electrodes. In the extreme, flooding of the electrodes or deposition of solid KOH in the electrode pores can cause a total loss of performance. Water removal from the cell is accomplished by evaporation into an excess flow of reactant gases. The rate of flow and moisture contents of the gases leaving the cell are measured and the former is adjusted as necessary to maintain the water balance. The excess flow rate can be determined from the inlet flow rate and the cell current. However, the determination of inlet flow rates is sometimes not highly accurate because the rotameter floats become sticky during operation. For this reason the exit flow rates are determined directly and with high accuracy by means of a Vol-u-Meter flow rate calibrator (G. H. Porter, Inc.). The moisture contents of the exit gases are calculated from dew point measurements made by impinging the gases on an electrically heated polished nickel sheet and observing the maximum temperature at which moisture condenses.

For operation on partially saturated gases the hydrogen and oxygen are sparged through water saturators whose temperatures are maintained equal to each other and constant to within 0.1°C. The saturator design was described previously[2].

3.3 Investigation of Materials

For operation at 100-200°C materials problems with at least some cell components will have to be solved. In particular, the selection of a suitable matrix is a major problem.

The matrix should have the following properties.

1. The ohmic resistance of the matrix, when loaded with KOH, should preferably be in the same range as asbestos, i.e., 0.025-0.050 ohms in a one-inch cell with 5N KOH at 70°C. However, for a limited lower portion of the current density range, 100-200 ma/cm^2, somewhat higher resistances could be tolerated.

2. The matrix should be stable enough in all concentrations of KOH at temperatures up to 200°C to give sustained performance for at least 1200 hours. If this degree of stability is not available at the start of the investigation, it is desirable then to start with a matrix which is stable for at least 1-2 hours. This matrix could be used for the investigation of operating variables while work continued to select a more stable matrix of comparable resistance.

3. To permit pressure operation, gas leakage through the matrix must be prevented. The matrix bubble pressure should safely exceed the minimum differential pressure (approximately 0.25 psi) which can be maintained by available controllers. Bubble pressure should therefore exceed 1 psig and as a practical matter should preferably be much higher.

4. The matrix must prevent electrode-electrode contact.

At the start of the investigation it was not obvious that any available material possessed all of the properties required either for the investigation of operating variables or for long term operation. However, such a material must be selected before any or all of the program tasks can be fulfilled completely. Consequently, the First Quarter effort concentrated on evaluating promising materials. These included (1) new commercial non-metallics, (2) asbestos, (3) PTFE-asbestos, (4) porous PTFE, , and (5) zirconia paper. The properties of these materials are given in Table 3-1.

The non-asbestos materials were given an initial screening by comparing their cell resistance and performance with that of the asbestos matrices. Data was obtained first at 70°C and 23% KOH because the greatest amount of data with asbestos matrices had been obtained previously under these conditions. Further screening was done at 100-200°C.

3.3.1 New Commercial Non-Metallics

In order to obtain information on new non-metallic materials, either commercial or semi-commercial, which might be suitable as a matrix, a literature search was made of the major abstracts of unclassified government contract work during the past 3-4 years. Several dozen reprints of these articles were ordered and received. None of these has revealed a new non-metallic material available in a form which could be used as a matrix in KOH at temperatures up to 200°C.

3.3.2 Asbestos

Asbestos matrices were investigated first. These included proprietary ACCO-I and ACCO-II Asbestos and Commercial Fuel Cell Asbestos (Johns Manville). Fuel Cell Asbestos is an Arizona chrysotile while ACCO-I Asbestos is made from a caustic-leached tremolite with 10% polyvinyl alcohol fiber as a binder. Both of these matrices have been described previously[1].

The ACCO-II Asbestos is made from a Canadian chrysotile asbestos and has no binder.

Table 3-1 shows the properties of these materials which pertain to their use as matrices. Because of its more open structure, ACCO-I Asbestos has a much lower bubble pressure (3 psi) than either ACCO-II Asbestos (20 psi) or Fuel Cell Asbestos (20 psi).

The suitability of Fuel Cell Asbestos as a matrix material at different temperatures was indicated from corrosion tests. After 2 hours immersion in 50-80% KOH, Fuel Cell Asbestos lost less than 1% of its weight at 100°C, at least 28% at 150°C and dissolved almost entirely at 200°C. Similarly ACCO-I Asbestos dissolved entirely in 80% KOH at 200°C. Consequently, no performance was obtained during several attempts to use Fuel Cell Asbestos as a matrix at 200°C. ACCO-II Asbestos gave highly unstable performance at 200°C within a few minutes after start-up. At 175°C, the stability of initial voltages was marginal.

Accordingly asbestos matrices are not suitable for investigating the effects of operating variables over the entire temperature range of 100-200°C. However, as shown in Table 3-2, asbestos can be used for obtaining initial performance data at temperatures up to 150°C. Comparing the 3 matrices at equal thickness, ACCO-I Asbestos gives the lowest resistance and highest peformance at atmospheric pressure. ACCO-II Asbestos gives somewhat higher resistance and lower performance.

The data of Table 3-2 on ACCO-I and Fuel Cell Asbestos were reported previously[1]. They are presented here to afford a comparison of the performance of asbestos matrices in sections 3.3.3 to 3.3.5.

Table 3-1

Matrix Properties

Matrix	Source	Status	Uncompressed Thickness Used* (Mils)	Available Void Volume (%)	Average Pore Size Microns	Bubble Pressure (psi)
ACCO-I Asbestos	American Cyanamid Co.	semi-commercial	20	87	Macroporous	3
ACCO-II Asbestos	American Cyanamid Co.	semi-commerical	20	72	"	20
Fuel Cell Asbestos	Johns Manville	commercial	20	62	"	20
PTFE -Asbestos	American Cyanamid Co.	experimental	12,24	65	"	< 0.2
PTFE Felt-TE 1029	American Felt Co.	commercial	60	72	"	"
PTFE Sheet 87-1	General Plastics Corp.	semi-commercial	11,30	67	4	"
PTFE Sheet W5125	Chemplast Inc.	semi-commercial	5	65	5	1
PTFE Sheet 233W5	Chemplast Inc.	semi-commercial	16	65	5	0.4
Proprietary PTFE Matrix A	American Cyanamid Co.	experimental	~ 60	80	Macroporous	"
Proprietary PTFE Matrix B	American Cyanamid Co.	experimental	4	45	20	20
Zirconia A Paper	H. F. Thompson	commercial	20	97	Macroporous	< 0.2

* One or more sheets

Table 3-2

Performance of Asbestos Matrices

Electrodes: AB-40
Pressure: 0 psig
Matrix Thickness: 20 Mils

Matrix	Temp. (°C)	KOH Conc. (%)	Cell Resistance (Ohms)	Working Voltage at Current Density (ma/cm^2) of: 0	100	200	300	400	500	600	800	1000
ACCO-I Asbestos	70	23	.025	1.05	.92	.87	.83	.78	.76	.74	.67	.55
Fuel Cell Asbestos	"	"	.050	1.03	.87	.82	.77	.72	.67	.62	.48	.35
ACCO-I Asbestos	100	50	.016	1.08	.98	.93	.90	.88	.85	.82	.76	.72
ACCO- II Asbestos	"	"	.032	1.10	.96	.91	.87	.83	.79	.75	.65	.52
Fuel Cell Asbestos	"	"	.037	1.08	.96	.91	.86	.81	.77	.73	.65	.57
ACCO-I Asbestos	140	68	----	1.12	1.02	.99	.95	.92	.88	.84	.76	.61
ACCO-II Asbestos	150	"	.027	1.14	1.03	.98	.94	.89	.85	.80	.68	.58

3.3.3 PTFE-Asbestos

The properties of a proprietary PTFE-asbestos matrix (containing 75% PTFE by weight) are given in Table 3-1. The available void volume is 65%. Bubble pressure is very low. The matrix has adequate strength at thicknesses as low as 12 mils.

The cell resistance and the performance obtained with this matrix at 70-200°C are shown in Table 3-3 and can be compared with that of asbestos matrices (Table 3-2). At 70°C the cell resistance was lower than that obtained with Fuel Cell Asbestos but higher than that obtained with ACCO-I Asbestos. Consequently, the performance was intermediate to that of the two asbestos matrices. At 100-140°C the cell resistance was higher and the performance was lower compared with that for the asbestos matrices. This may be due to a certain lack of reproducibility in early samples of the PTFE-Asbestos matrix.

At 200°C and 75-80% KOH, low cell resistance and very high performance were obtained with the PTFE -asbestos matrix. This was the first performance to be obtained with AB-40 electrodes at temperatures of 200°C and is higher than that obtained previously at any lower temperature and KOH concentration. The performance of the run marked with the asterisk is the highest obtained to date with these electrodes at 100-300 ma/cm^2 and atmospheric pressure (1.07 and 1.00 volts at 100 and 300 ma/cm^2 respectively). This further confirms the conclusion reported previously[1] that cell performance generally increases with increasing temperature and KOH concentration and demonstrates the advantage to be gained in operating a matrix fuel cell at temperatures as high as 200°C, (approximately 50 mv and 100 mv compared to the performance at 150° and 100°C) provided that a suitable matrix is developed.

Table 3-3

Performance of PTFE Asbestos Matrix

Cell: One Inch
Electrodes: AB-40
Pressure: 0 psig

Thickness (Mils)	Temp. (°C)	KOH Conc. (%)	Cell Resistance (Ohms)	Working Voltage at Current Density (ma/cm²) of:								
				0	100	200	300	400	500	600	800	1000
40	70	23	.037	1.09	.90	.84	.80	.74	.69	.63	-	-
22	100	50	.067	1.09	.94	.85	.78	.71	-	.54	-	-
24	"	"	.057	1.07	.95	.88	.80	.71	-	-	-	-
22	140	65	.065	1.07	.98	.91	.83	.72	-	-	-	-
12*	200	75	.025	1.11	1.07	1.04	1.00	.96	.93	.89	.82	.76
12	"	75	.025	1.10	1.07	-	-	-	-	-	-	-
24	"	80	.040	1.15	1.08	1.02	.97	.92	.87	.82	.73	.64

*Highest Performance to date of AB-40 Electrodes at 100-300 ma/cm² and atmospheric pressure.

3.3.4 Porous PTFE

Based on resistance to KOH corrosion, PTFE is one of the few non-metallics which is potentially suitable as a matrix material at temperatures up to 200°C. As far as can be ascertained at present, only 3 manufacturers offer commercial or semi-commercial quantities of thin porous PTFE which might be used as a matrix. In addition, two experimental proprietary PTFE matrices (designated A and B), developed prior to the start of the contract, are available. The properties of these matrices are listed in Table 3-1.

A major problem in using porous PTFE as a matrix is that PTFE gives very high cell resistance because it is not wet by the electrolyte. To obtain complete wetting (i.e., zero contact angle), the electrolyte surface tension should be below the critical surface tension of PTFE. However, the critical surface tension of PTFE is only 18 dynes/cm[2] while the surface tension of KOH solutions is considerably higher, e.g., 85 dynes/cm for 30% KOH at 20°C. The critical surface tension of PTFE can be increased to approximately 30 dynes/cm by etching[2], for example with Tetra-Etch, a super-saturated solution of a sodium aryl compound in highly polar solvents (W. L. Gore and Associates). This treatment increased the wettability of PTFE somewhat, but for complete wettability, it was necessary to add wetting agents which drastically reduce the surface tension of the electrolyte. A search for suitable commercially-available, non-volatile wetting agents which are stable in all concentrations of KOH at temperatures up to 150-200°C showed that two perfluorinated surfactants designated FC-95 and FC-128 (3M Company) offer the most promise. Data provided by the 3M Company show that FC-95 and FC-128 at concentrations of < .1% reduce the room temperature surface tension of 30% KOH to 25 and 17 dynes/cm respectively. Consequently, at these KOH concentrations, FC-95 should produce complete wetting of etched PTFE while FC-128 should produce complete wetting of both etched and unetched PTFE.

unetched PTFE. . Surface tension data for these wetting agents in higher concentrations of KOH and at higher temperatures are not available at present.

3.3.4.1 PTFE Felt

PTFE Felt TE-1029 (American Felt Co.) is relatively thick (60 mils) but has a high available void volume (72%). The felt is easily compressed inside or outside of the cell to a 20-30 mil thickness and a void volume of 50%. Either uncompressed or compressed, its bubble pressure is very low.

Table 3-4 shows the cell resistance and performance of the PTFE felt matrix, both unetched and etched. With no wetting agent in the KOH, the unetched matrix produced high cell resistance, 17 and 3 ohms at 70°C and 100°C respectively, and gave no performance under load. The addition of only 0.01% of FC-95 to the KOH increased the KOH absorption by the matrix nearly 4 fold from 0.14 to 0.51 ml KOH/ml dry matrix, and reduced the cell resistance to 0.087 ohms. However, no performance was obtained due possibly to flooding of the cell. With somewhat lower KOH loadings, 0.24-0.31 ml/ml, performance under load was obtained at both 70°C and 100°C. At both temperatures the cell resistance (0.065 - 0.076 ohms), though considerably lower than with no wetting agent present, was still higher than that of either Fuel Cell Asbestos or ACCO Asbestos. Consequently, cell performance was generally lower than with either of the asbestos matrices (Table 3-2). Etched PTFE felt gave higher cell resistance (0.09-0.13 ohms) and lower performance at 70°C than the unetched felt even though it was wet completely by 23% KOH containing either FC-95 or FC-128. At least part of the increase in cell resistance was probably due to shrinkage and thickening of the matrix caused by etching.

TABLE 3-4

PERFORMANCE OF PTFE MATRICES

Electrodes: AB-40
Gas Pressure: 0 psig

Matrix	Temp. (°C)	KOH Conc. (%)	Wetting Agent	Cell Assembly Pressure (psi)	Cell Resistance (Ohms)	Working Voltage at Current Density (ma/cm^2) of: 0	40	100	200	300	400	500	600	800	1000
PTFE Felt (Un-etched)	70	23	None		17	.93	0	-	-	-	-	-	-	-	-
	70	23	.01% FC-95	180	.076	1.09		.87	.78	.70	.60	-	-	-	-
PTFE Felt(Unetched)	100	50	None		3	.98									
PTFE Felt (Unetched)	100	50	.01% FC-95	180	.065	1.06		.88	.68	-	-	-	-	-	-
PTFE Felt[1] (Etched)	70	23	.01% FC-95	180	.09	1.07	.90	.76	-	-	-	-	-	-	-
PTFE Felt[1] (Etched)	70	23	.01% FC-95	180	.13	1.07	.90	.78	-	-	-	-	-	-	-
PTFE Felt[1] (Etched)	70	23	.1% FC-128	180	.12	1.06	.90	.79	-	-	-	-	-	-	-
PTFE Felt[1] (Etched)	70	23	.1% FC-128	180	.13	1.07	.89	.68	-	-	-	-	-	-	-
Proprietary Matrix (A) (Un-etched)	70	23	FC-128[7]	180	.060	-	-	.85	.79	.71	.65	-	-	-	-
Proprietary Matrix (A) (Etched)	70	23	.01% FC-95	180	.036	1.05	.92	.88	.84	.80	.76	.72	.68	.59	-
Proprietary Matrix (A) (Etched)	200	75	.01% FC-95	180	.051	1.04	-	.99	.96	.92	.88	.82	.77	.64	.51
87-1[2] (Etched)	70	23	.01% FC-95	180	.0023	0[5]	-	-	-	-	-	-	-	-	-
87-1[2] (Etched)	70	23	.01% FC-95	60	.005	0[5]	-	-	-	-	-	-	-	-	-
87-1[2] (Etched)	70	23	.1% FC-128	10	.57	-	-	-	-	-	-	-	-	-	-
87-1[2] (Etched)	70	23	.1% FC-128	20	.009	0[5]	-	-	-	-	-	-	-	-	-
87-1[2] (Sintered)	70	23	.01% FC-95	60	.023	0[5]	-	-	-	-	-	-	-	-	-
W 5125[3] (5 mil)	70	23	.1% FC-128	180	.009	0[5]	-	-	-	-	-	-	-	-	-
W 5125[3] (10 mil)[4]	70	23	.1% FC-128	180	.018	0[5]	-	-	-	-	-	-	-	-	-
W 5125[3] (10 mil)[4]	70	23	.1% FC-128	16	.007	0[5]	-	-	-	-	-	-	-	-	-
233 WS[3]	70	23	.1% FC-128	120	.050	1.09	-	.88	.82	.77	.72	-	.63	.51	.39
233 WS[3]	200	75	.01% FC-95	30	.0073	0[5]	-	-	-	-	-	-	-	-	-
233 WS[3]	200	75	.01% FC-95	15	.015	0[5]	-	-	-	-	-	-	-	-	-
Proprietary Matrix (B) (4 mils)	70	23	.01% FC-95	180	.0032	0[5]	-	-	-	-	-	-	-	-	-
Proprietary Matrix (B) (4 mils)	70	23	.01% FC-95	60	.005	0[5]	-	-	-	-	-	-	-	-	-
ACCO-I Asbestos[6]	70	23	None	180	.025	1.05	-	.92	.87	.83	.78	.76	.74	.67	.55
	"	"	.01% FC-95	180	.025	1.07	-	.92	.88	.84	.79	.76	.73	.66	.59

(1) American Felt Company.
(2) General Plastics Company.
(3) Chemplast, Inc.
(4) Two layers of 5 mil sheet.
(5) Electrodes broke through matrix.
(6) Included to show effect of FC-95

The wetting agent itself did not affect cell performance. This can be seen from the nearly identical performance of runs employing an ACCO-I Asbestos matrix with and without FC-95 in the KOH. (Table 3-4). Furthermore after 600 hours of operation at 70°C and 100 ma/cm^2, the voltage of a cell with ACCO-I Asbestos and FC-95 in the KOH decreased by only 20 mv. Thus at 70°C, at least, FC-95 does not appear to poison the electrodes or cause them to flood.

3.3.4.2 Proprietary PTFE Matrices

Proprietary matrix (A) has a high void volume (80%) but a very low bubble pressure. Cell resistance and performance with this matrix are shown in Table 3-4. In the unetched form the matrix wet by 23% KOH containing FC-128 yielded higher cell resistance (0.060 ohm) and lower cell performance than the asbestos matrices. However, when used in the etched form with FC-95 in the KOH, both cell resistance (0.036 ohm) and performance were intermediate to those of ACCO-I and Fuel Cell Asbestos. This is the best performance obtained to date in this work with an all-PTFE matrix and demonstrates the possibility of good performance with a matrix of thin porous PTFE when it is completely wetted. At 200°C and 75% KOH the cell resistance was 0.057 ohm and performance was obtained up to 1000 ma/cm^2 but was not as high as reported in Section 3.3.3 for the proprietary PTFE-asbestos under these conditions.

Proprietary matrix (B) has a high bubble pressure (20 psi) but only 40-55% voids. To avoid the high cell resistance generally associated with this low void volume, very thin sheets (4 mils) were used. However, the electrodes broke through these sheets and no performance was obtained.

3.3.4.3 Commercial PTFE Matrices

The performance of commercial porous Teflon sheets as matrices is also shown in Table 3-4.

In tests with etched General Plastics Sheet 87-1 (11 mils thick) the electrodes repeatedly broke through the matrix and short-circuited the cell. Breakthrough occurred at cell assembly pressures of 20-180 psi. At 10 psi, the matrix did not break, but cell resistance was extremely high (0.57 ohm). Consequently, these sheets are not suitable.

Electrode breakthrough also occurred with Chemplast Sheets W5125 (5 mils) and, at high temperatures, with Chemplast 233WS (16 mils). However, a 16 mil sheet, unetched but completely wetted by 23% KOH containing FC-95 gave cell resistance (0.050 ohm) and performance identical to that of Fuel Cell Asbestos at 70°C. This is the highest performance obtained to date in this work with a commercial porous PTFE. At 200°C breakthrough occurred even at assembly pressures as low as 15 psi. Interposing a fine 80 mesh screen between each electrode and the matrix did not prevent breakthrough. Sintering the sheet at 330°C before use prevented breakthrough but gave very high cell resistance.

3.3.5 Zirconia Paper

Zirconia paper (H. I. Thompson Company) is made from zirconia fiber containing 4-5% Calcia, and 5% organic binder. The paper has an extremely high void volume (approximately 96%) and absorbs six times its own weight of 23% KOH. The paper is 18 mils thick but compresses considerably within the cell. It was found necessary to use at least three sheets of the paper to prevent electrode breakthrough. The bubble pressure is very low.

The zirconia paper was of interest because it yielded substantially lower cell resistance (0.012-0.019 ohm) than any of the other matrix materials. Consequently it was found to be capable of giving higher initial performance than any of the other matrices at current densities above 300 ma/cm^2 at temperatures up to 200°C. However, from the results of a number of runs over a range of temperatures and electrolyte loading, the initial performance was not found to be reproducible. Those runs which gave the best and most reproducible performance are shown in Table 3-5. The performance of the run at 200°C marked with the asterisk is the highest obtained to date with AB-40 electrodes at 300-1000 ma/cm^2 and atmospheric pressure (1.00 and 0.80 v at 300 and 1000 ma/cm^2). However, at 100 ma/cm^2, the cell voltage (1.02-1.04v) was substantially below that obtained with the PTFE -asbestos matrix (1.07 v) even though the cell resistance of the latter (.025 ohm) was somewhat higher (Table 3-3). This phenomenon and the non-reproducibility of performance were probably caused by gas leakage through the matrix. The degree of this leakage might vary with the arrangement of pin holes in the adjacent sheets. Increasing the number of sheets from 3 to 4 did not noticeably improve reproducibility.

Consequently, although zirconia paper demonstrated further the extremely high performance possible with these electrodes at temperatures up to 200°C, it was not suitable for obtaining reproducible initial performance data.

Beaker corrosion tests at 70% KOH showed that after three days at 150°C, the zirconia paper disintegrated into individual fibers because the binder dissolved. Nevertheless, the fibers remained intact. After three days at 200°C, the fibers themselves dissolved. Thus a zirconia matrix made without a binder and in a form which prevented gas leakage might be suitable for high long term performance at temperatures up to 150°C but not much higher.

Table 3-5

Performance of Zirconia Paper Matrix

Electrodes: AB-40
Pressure: 0 psig

Temp. (°C)	KOH Conc. (%)	Cell Resistance (Ohms)	Working Voltage at Current Density (ma/cm^2) of: 0	100	200	300	400	500	600	800	1000
70	23	.016	1.06	.90	.87	.84	.82	.80	.78	.75	.70
100	45	.016	1.07	.97	.93	.90	.87	.84	.81	.73	.64
"	"	.012	1.07	.96	.93	.90	.88	.86	.83	.77	.70
150	68	.014	1.06	1.02	.99	.97	.94	.91	.87	.79	.70
200*	75	.018	----	1.04	1.02	1.00	.98	.95	.92	.86	.80
"	"	.012	----	1.02	1.01	1.00	.98	.95	.92	.85	.78
"	"	.012	----	1.02	1.01	.99	.97	.95	.92	.86	.79
"	"	.014	----	1.03	1.02	.99	.97	.94	.91	.84	.76
"	"	.019	----	1.03	1.01	.99	.97	.95	.92	.84	.75

*Highest performance to date of AB-40 electrodes at 300-1000 ma/cm^2 and atmospheric pressure.

3.4 Investigation of Operating Variables

The effects of operating variables on initial performance are to serve together with long term test results as a basis for recommending preferred operating conditions. It is anticipated that these preferred conditions will be recommended from the following considerations. (1) maximum performance, (2) highest temperature level at which long term stability can be obtained, (3) operation with or without pressure, and (4) minimum gas recycle, i.e., minimum parasitic power and weight for gas recycle.

The desired ranges of operating variables, stated in section 1.2 are listed below:

Current Density:	100 - 1000 ma/cm^2
Temperature:	100 - 200°C
Pressure:	0 - 60 psig
Electrolyte (KOH) Concentration:	30% by weight to the solubility limi
Electrolyte Loading:	Variable, depending on matrix
Reactant Moisture Content:	0-90% saturated with respect to cell conditions
Fraction of Product Water Removed at Each Electrode:	0-1

With the aid of the Statistical Analysis Group, an experimental design was devised to give maximum information about the effects of these operating variables on initial performance with a reasonable number of runs. However, the matrix materials investigation showed that matrices available at present are unsuitable for carrying out the entire design and that a choice must be made between investigating the operating variables at temperatures up to 200°C at atmospheric pressure only or at temperatures up to 150°C over the range of pressures specified above. The latter option was chosen because:

1. It does not require the elimination of an important variable.

2. Sustained long term performance is more likely to be achieved in this temperature range.

3. In the course of investigating matrix materials, the general effects of temperature and KOH concentration at 150-200°C have already been observed. These effects, together with the pressure effects to be determined at 100-150°C, should permit a reasonably accurate estimate of the performance to be expected at 150°C-200°C at pressures up to 60 psig.

Accordingly, a second experimental design was devised for the temperature range 100-150°C (Table 3-6). This design permits a 3-dimensional mapping of the temperature-pressure-KOH concentration effects together with a determination of the precision of the data. It includes all limiting combinations of these variables (e.g., 150°C, 60 psig 75% KOH) together with intermediate combinations. A "center" point is to be run 5 times. In all runs, cell voltage will be determined at 0-1000 ma/cm^2 in increments of 100-200 ma/cm^2. Each run will be made separately with fresh electrodes, and 2-3 polarization curves will be determined consecutively. This will permit a determination of the effect of operating variables on the boost in performance which results from consecutive running of polarization curves to high current densities (section 3.2.1). The remaining two variables, reactant moisture content and fraction of product water removed at each electrode, will be studied at preferred combinations of temperature, pressure, and KOH concentration. They are not included in a general 5-variable design because previous work indicates little probability that they will have a significant effect on initial performance (although they do affect sustained performance). Furthermore, such a design would require either an inordinate number of runs or considerably less information concerning the effects of temperature, pressure, and KOH concentration.

Table 3-6

Experimental Design for Initial Performance Data (100-150°C)

Basic 3-Variable Design

Temp. (°C)	Pressure (psig)	KOH Conc. (%)	KOH Solubility (%)
100	0	30, 60	65
100	15	45	65
100	30	45	65
100	60	30, 60	65
125	15	30, 45*, 60	70
125	30	30, 45, 60	70
150	0	55, 65, 75	80
150	15	65	80
150	30	45, 55, 75	80
150	60	30, 55, 75	80

*Run 5 Times

Additional 2-Variable Runs Under Preferred Conditions

Fraction of Water Removed at Each Electrode: 0, 1/3, 2/3, 1

Reactant Moisture Content: 0, 50, 90% Saturated with Respect to Cell Conditions

ACCO-II Asbestos was chosen as the matrix material for this design because of its combination of high bubble pressure and low cell resistance. Experimental work has begun.

3.5 Life Testing Results

From the definition of stable long term performance (section 1.2), the overall voltage decline rate should not exceed 4.2 mv/100 hrs. during a 1200 hour period. Life testing was started with Fuel Cell Asbestos and ACCO-I Asbestos matrices at 100-125°C to determine the maximum temperature suitable for the long term stability of asbestos matrices and to identify the factors causing performance losses. Operating variables included the current density, the KOH concentration, and the moisture content and H_2/O_2 ratio of the inlet gas streams. Runs at KOH concentrations above 50% were made by concentrating 50% KOH in the cell while under load by setting the gas flows for water balance at the desired concentration. Overall voltage decline rates were generally calculated from the average slopes of the voltage-time curves.

3.5.1 Initial and Maximum Voltage

Life test results at 100°C and 125°C are summarized in Tables 3-7 and 3-8 respectively. The tables list initial, maximum, and present (or final) voltages. The "initial" voltage is the voltage observed when the desired current is first drawn and the KOH concentration is the same as was loaded into the matrix. In runs where the KOH was further concentrated within the cell, the initial voltage generally rose by 25-45 mv to a maximum level. Nearly all of this rise occurred within 30 minutes i.e., within the time required to concentrate the KOH to the desired level. Accordingly the "maximum" voltage is the significant voltage at the start of the test, unless the maximum occurred after a period considerably greater than 30 minutes.

Table 3-7

Life Tests-100°C

Life Test No.	Matrix	Current Density (ma/cm²)	KOH Conc. (WT. %)	Initial Electrode Condition	Inlet Gas Condition	Inlet Flow Ratio (H_2/O_2)	Test Duration (Hrs.)	Working Voltage			Overall voltage decline rate (mv / 100 hrs.)	Cell Resistance (Milliohms)			Status	Reason for Termination
								initial	maximum	now or final		initial	minimum	now or final		
2-163	Fuel Cell Asbestos	100	35	Dry	Humidified at 75°C	1.0	664	.906	.906	.855	7.7	8.2	8.2	12.3	Terminated	(a)
2-159	" " "	"	44	"	Humidified at 60°C	1.0	737	.924	.924	.861	8.5	6.6	6.2	11.8	"	"
2-167	" " "	"	50	"	Dry	1.1	232	.919	.924	.840	36	9.0	9.0	16.7	"	"
2-168	" " "	"	"	"	Dry	1.1	160	.918	.921	.882	24	10.0	10.0	11.0	"	"
2-171	" " "	"	"	"	Humidified at 40°C	1.0	329	.902	.925	.873	16	11.4	8.6	13.2		
2-172	ACCO-I Asbestos	100	30	Dry	Dry	1.1	523	.943	.943	.920	4.4	3.8	3.8	6.4	"	"
2-176	" "	"	44	"	"	1.2	476	.918	.924	.888	7.6	4.6	4.6	7.9	"	"
2-185	" "	"	"	"	Humidified at 60°C	1.1	259	.901	.922	.904	7.0	6.5	4.3	5.5	"	"
2-175	" "	"	50	"	Dry	1.1	497	.938	.940	.916	4.8	5.8	4.2	6.0	"	"
2-180	" "	"	"	"	Humidified at 40°C	1.1	666	.908	.946	.889	8.6	5.8	5.1	6.3	"	"
2-186	" "	"	"	"	" " "	1.0	450	.930	.936	.913	5.1	4.9	4.9	6.4	"	"
2-181	" "	"	"	"	Humidified at 50°C	1.0	641	.930	.940	.902	5.9(a)	5.5	4.8	6.7	"	"
2-196	" "	"	"	Wet	Dry	1.1	500	.949	.957	.946	0.6	4.0	3.0	3.2		
2-207	" "	"	"	"	"	1.1	690	.962	.962	.947	2.2	3.8	2.7		Continuing	--
2-193	ACCO-I Asbestos	200	50	Dry	Dry	1.0	282	.880	.880	.843	13	6.0	6.0	6.7	Terminated	(a)
2-202	" "	"	"	Wet	"	0.67	813	.926	.926	.900	3.2	3.8	2.8	3.0	Continuing	--
2-208	" "	300	"	"	"	1.0	1050	.892	.899	.848	4.2(a)	3.6	2.7	3.0	"	--
2-212	" "	400	"	"	"	1.0	114	----	.911	.838	65	3.0	3.0	3.0	Continuing	--

(a) Based on initial voltage

Table 3-8

Life Tests-125°C

TLT No.	Matrix	Current Density (ma/cm²)	KOH Conc. (WT. %)	Initial Electrode Condition	Inlet Gas Condition	Inlet Flow Ratio (H_2/O_2)	Test Duration (Hrs.)	Working Voltage			Overall voltage decline rate $\left(\frac{mv}{100\ hrs.}\right)$	Cell Resistance (Milliohms)			Status	Reason for Termination
								initial	maximum	now or final		initial	minimum	now or final		
2-195	ACCO-I Asbestos	100	60	Dry	Dry	1.0	162	.940	.955	.880	46	4.2	4.2	7.2	Terminated	(b)
2-201	" "	"	"	"	"	1.0	237	.948	.973	.918	23	4.0	4.0	4.6	"	"
2-194	" "	"	"	Wet	"	1.0	429	.940	.985	.862	28	4.5	3.8	5.5	"	(a)
2-198	" "	"	"	"	"	1.0	618	.918	.985	.905	13	3.8	3.5	4.2	Continuing	--
2 209	" "	"	"	"	"	0.67	332	.930	.972	.928	13	4.0	4.0	4.0	Terminated	(a)
2-200	" "	"	"	"	Humidified at 45°C	1.0	452	.802	.978	.855	27	3.5	3.5	6.2	"	"
2-206	" "	"	"	"	Humidified at 50°C	1.0	521	.950	.995	.830	32	4.2	3.7	4.5		
2-211	ACCO-II Asbestos	"	"	"	Dry	1.0	330	.939	.989	.933	17	4.2	4.2	4.2	Continuing	--

(a) Decline rate defined

(b) Gas cross-leakage

As discussed in section 3.2.1, previous work (with electrodes which were dry prior to cell assembly) showed that the cell voltage increased by 20-40 mv at current densities up to 500-600 ma/cm^2 if a polarization curve is first run out to higher current densities (600-1000 ma/cm^2). Since this procedure was generally not followed in starting the life tests, the maximum voltages for runs with dry electrodes are approximately 40 mv below the voltages possible with this procedure.

In some runs the electrodes were pre-wet with KOH solution before they were assembled in the cell, either by immersion for 1-3 days or by vacuum immersion for 5 minutes. It was found that pre-wetting the electrodes increased the maximum voltage by 20-45 mv (Tables 3-7 and 3-8).

In one run (TLT-2-207) an attempt was made to further increase the voltage of pre-wet electrodes by first running a polarization curve out to 600 ma/cm^2. As shown in Table 3-7, the voltage did not increase. This indicates that the voltage increases produced by high current densities with "dry" electrodes may be due to a wetting effect.

3.5.2 100°C Tests

A summary of life tests conducted at 100°C is given in Table 3-7. Figures 3-2 through 3-4 show in detail the change of cell voltage and resistance with time at current densities of 100-400 ma/cm^2.

With 20 mil Fuel Cell Asbestos, an average maximum voltage of 0.92v was obtained at 100 ma/cm^2. High overall voltage decline rates (8-36 mv/100 hrs. were encountered, caused partly by a continual rise in cell resistance (Figure 3-

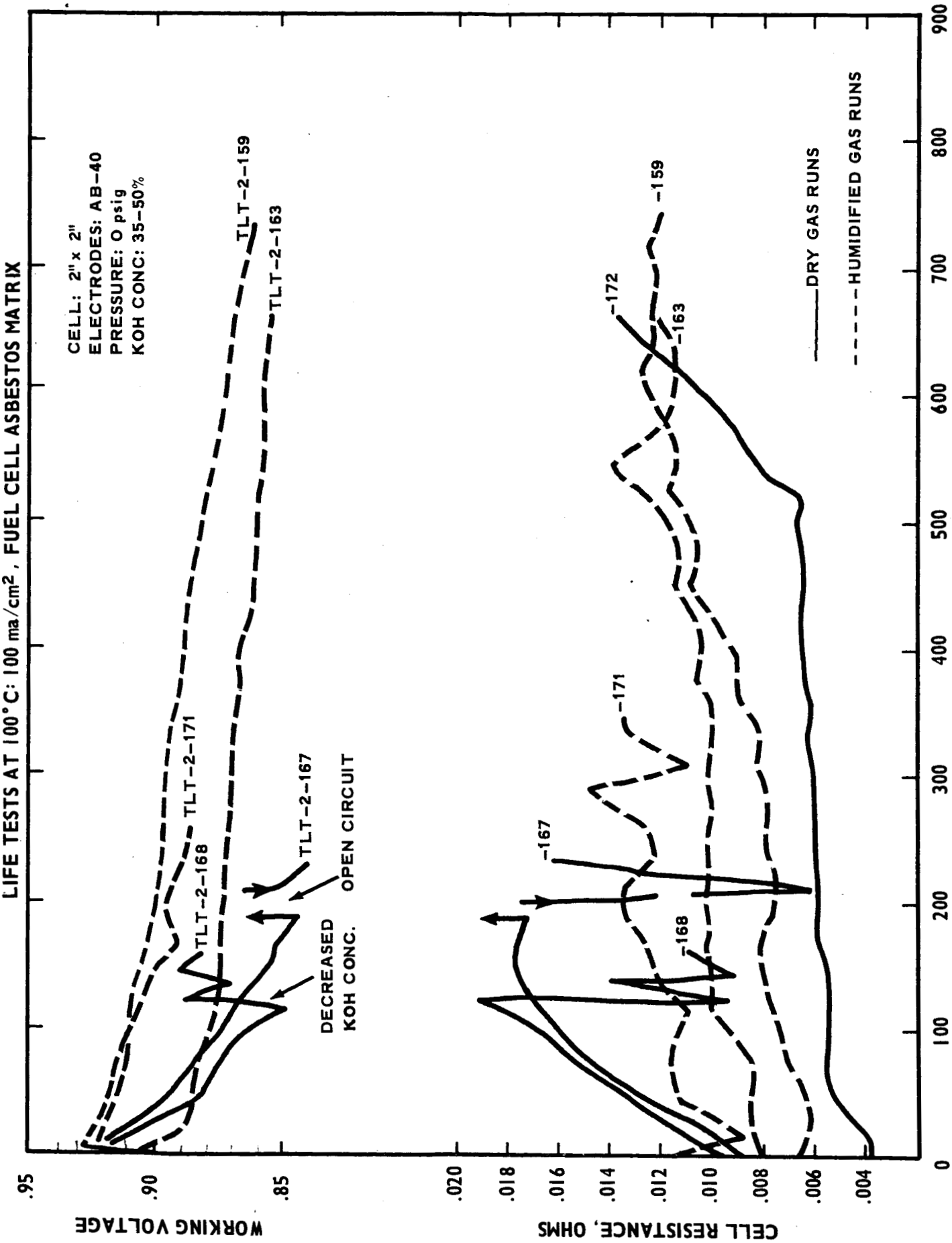
LIFE TESTS AT 100°C: 100 ma/cm², FUEL CELL ASBESTOS MATRIX
CELL: 2" x 2"
ELECTRODES: AB-40
PRESSURE: O psig
KOH CONC: 35-50%
TLT-2-159
TLT-2-163
TLT-2-171
TLT-2-168
TLT-2-167
DECREASED KOH CONC.
OPEN CIRCUIT
-172
-163
-159
-171
-167
-168
DRY GAS RUNS
HUMIDIFIED GAS RUNS
WORKING VOLTAGE
CELL RESISTANCE, OHMS
TIME, HOURS
.95
.90
.85
.020
.018
.016
.014
.012
.010
.008
.006
.004
0
100
200
300
400
500
600
700
800
900

FIGURE 3-2

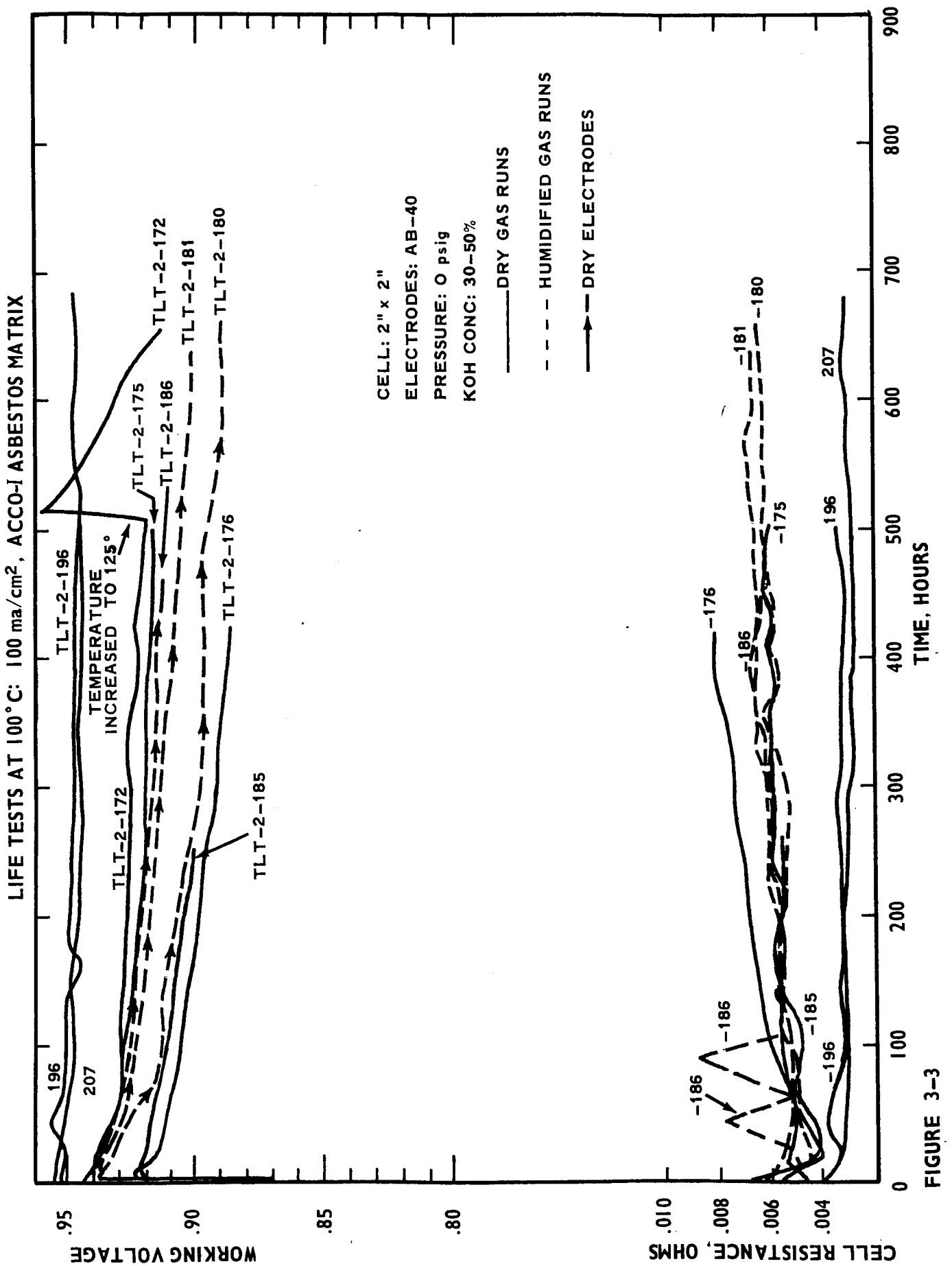
LIFE TESTS AT 100°C: 100 ma/cm², ACCO-I ASBESTOS MATRIX
CELL: 2" x 2"
ELECTRODES: AB-40
PRESSURE: O psig
KOH CONC: 30-50%
DRY GAS RUNS
HUMIDIFIED GAS RUNS
DRY ELECTRODES
TEMPERATURE INCREASED TO 125°
TLT-2-196
TLT-2-172
TLT-2-175
TLT-2-186
TLT-2-181
TLT-2-180
TLT-2-176
TLT-2-185
196
207
-176
-186
-175
-181
-180
-185
WORKING VOLTAGE
CELL RESISTANCE, OHMS
TIME, HOURS

FIGURE 3-3

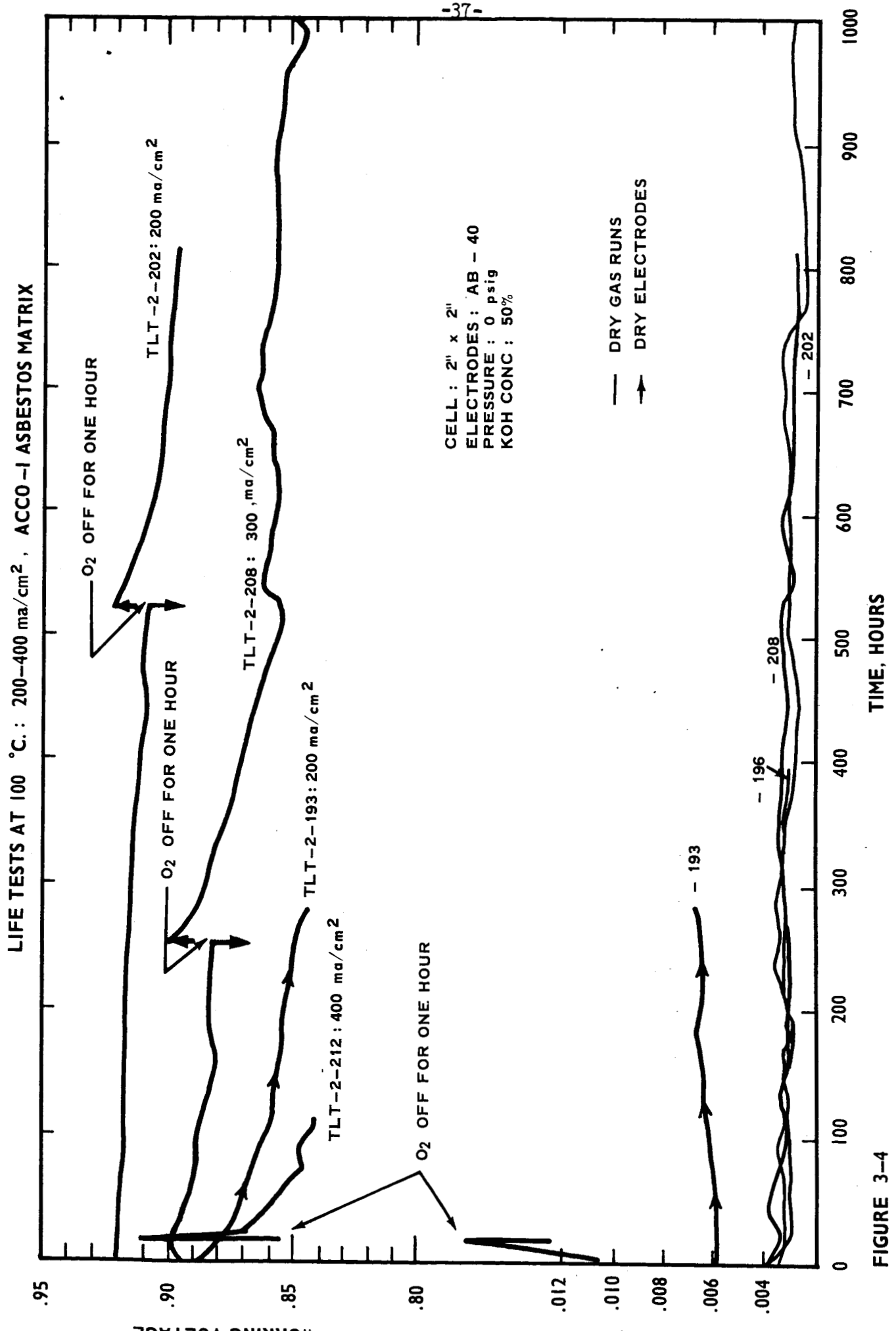
LIFE TESTS AT 100 °C. : 200–400 ma/cm², ACCO–I ASBESTOS MATRIX
O2 OFF FOR ONE HOUR
TLT–2–202: 200 ma/cm²
O2 OFF FOR ONE HOUR
TLT–2–208 : 300 ,ma/cm²
TLT–2–193: 200 ma/cm²
TLT–2–212 : 400 ma/cm²
O2 OFF FOR ONE HOUR
CELL : 2" x 2"
ELECTRODES : AB – 40
PRESSURE : 0 psig
KOH CONC : 50%
DRY GAS RUNS
DRY ELECTRODES
– 193
– 196
– 208
– 202
WORKING VOLTAGE
RESISTANCE, OHMS
TIME, HOURS
.95
.90
.85
.80
.012
.010
.008
.006
.004
0
100
200
300
400
500
600
700
800
900
1000

FIGURE 3–4

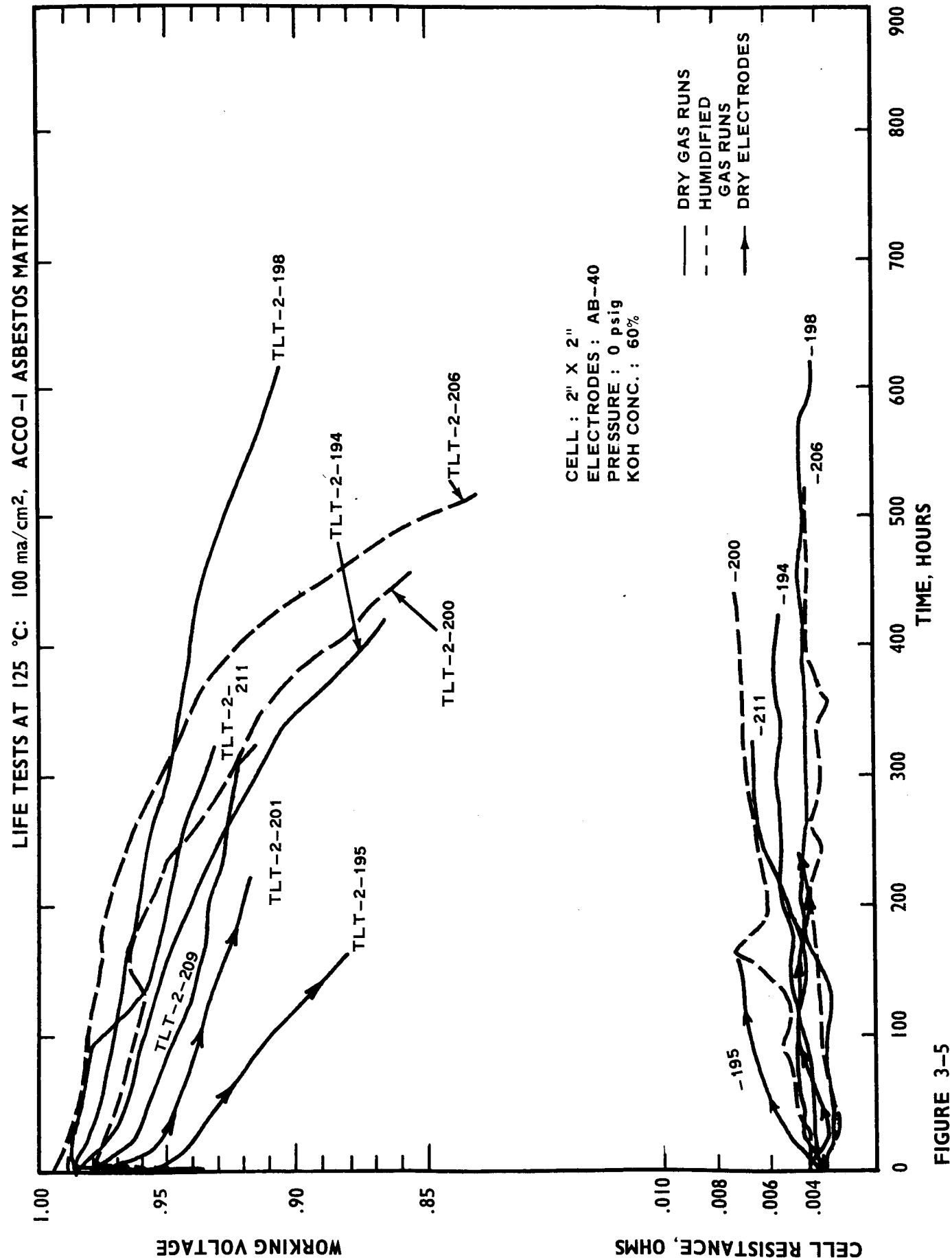
LIFE TESTS AT 125 °C: 100 ma/cm², ACCO-I ASBESTOS MATRIX
CELL : 2" X 2"
ELECTRODES : AB-40
PRESSURE : 0 psig
KOH CONC. : 60%
DRY GAS RUNS
HUMIDIFIED GAS RUNS
DRY ELECTRODES
TLT-2-198
TLT-2-194
TLT-2-206
TLT-2-200
TLT-2-211
TLT-2-201
TLT-2-209
TLT-2-195
-198
-206
-200
-194
-211
-195
WORKING VOLTAGE
CELL RESISTANCE, OHMS
TIME, HOURS
1.00
.95
.90
.85
.010
.008
.006
.004
0
100
200
300
400
500
600
700
800
900

FIGURE 3-5

At 50% KOH, cell voltage was more stable with humidified gases than with dry gases (Runs TLT-2-167, -168 and -171). With humidified gases, at constant inlet gas rates, the cell voltage was more stable at 35-44% KOH than at 50% (Runs TLT-2-163, -159 and -171 respectively). Since the overall voltage decline rates were considerably higher, even at 100°C, than the maximum desired in this work and since ACCO-I Asbestos gives higher initial performance, life testing of Fuel Cell Asbestos was discontinued in favor of ACCO-I Asbestos.

With ACCO-I Asbestos (Figure 3-3), an average maximum voltage of 0.94 v was obtained at 100 ma/cm^2 when the electrodes were dry prior to assembly in the cell. Operation on dry gases at 100 ma/cm^2 generally gave an overall voltage decline rate in the range 3-5 mv/100 hrs. accompanied by slight increases in cell resistance. This decline rate was substantially lower than that of Fuel Cell Asbestos, both on dry and humidified inlet gases. Unlike Fuel Cell Asbestos, the voltage stability of ACCO-I Asbestos was not improved by substituting humidified gases for dry gases. This may be due to the higher KOH mobility in the ACCO Asbestos matrix. Higher mobility tends to serve the same function as does humidifying the inlet gases in minimizing KOH concentration gradients which cause voltage losses.

It was found that pre-wetting the electrodes improved the voltage stability. By this means, satisfactory voltage decline rates as low as 0.6 - 2.2 mv/100 hrs. were obtained during a 500 hour period (TLT-2-196, and -207 respectively). Pre-wetting the electrodes increased the average maximum voltage to 0.96.

Figure 3-4 shows the results of life tests on dry gases at 200-400 ma/cm^2. At 200 ma/cm^2, the voltage decline rate was reduced from 13 to 3.2 mv/100 hrs. during a period of 800 hours by pre-wetting the electrodes and possibly by reducing the H_2/O_2 flow ratio (Runs TLT-2-193, and -202). At 300 ma/cm^2, the voltage decline rate was 4.2 mv/100 hours during

1050 hours, (TLT-2-208). Thus, at 100°C and current densities of 100-300 ma/cm^2, satisfactory overall voltage decline rates were achieved for periods of 500-1000 hours. (The cells were not run for 1200 hours because of the need to begin life testing at higher temperatures.) This level of voltage stability has not yet been achieved at 400 ma/cm^2 where the voltage decline rate in one run (TLT-2-212) was 65 mv/100 hours.

3.5.3 125°C Tests

The results of life tests at 125°C and 60% KOH are given in Table 3-8 and Figure 3-5. The average maximum voltage was 0.98 v.

The voltage declined much faster than at 100°C (Figure 3-3). Furthermore, the voltage decline at 125°C accelerated after 300-400 hours whereas at 100°C this rate was nearly linear. With dry gases and dry electrodes, the decline rate was 23-46 mv/100 hours (runs TLT-2-195 and -201). Pre-wetting the electrodes reduced this to 13-28 mv/100 hours (TLT-2-194, -198, -209, and -211). No further reduction was obtained by humidifying the inlet gases at 45-50°C (TLT-2-200 and -206). The voltage decline rate of 28 mv/100 hours occurred at a KOH loading of 3 g/g dry matrix and an H_2/O_2 flow ratio of 1.0 (TLT-2-194). Increasing the KOH loading to 4.0 g/g (TLT-2-198) or decreasing the H_2/O_2 flow ratio to 0.67 (TLT-2-209) decreased the voltage decline rate to 13 mv/100 hours. In nearly all of these tests, the cell resistance did not rise appreciably (Figure 3-5) indicating that the voltage decline was not due to any overall flooding or drying out of the electrodes.

In the two runs with dry electrodes loaded into the cell (TLT-2-195 and -201) at least part of this decline was caused by cross-leakage of gas through the matrix. Cross-leakage was detected directly from measurements of the exit gas rates. Furthermore, since it increased water production by the direct combination of hydrogen and oxygen, cross-leakage was evidenced by dew point measurements of the exit gases which showed the KOH concentration to be abnormally low.

3.5.4. Evaluation of Used Electrodes

It would be desirable to determine whether the cell voltage loss after prolonged operation results from a loss of electrode activity. In principle, this can be done by washing and then drying the electrodes at the end of a life test, assembling them in a fresh cell, and comparing their polarization with that of unused electrodes. However, it is difficult to obtain unambiguous results since some of the asbestos adheres firmly to the electrodes and it is extremely difficult to remove all of this asbestos without removing some of the platinum as well. The difficulty of removal increases with the test time. The extent of the problem is illustrated in the polarization data of Figure 3-6. Two sets of 1" discs were cut from the used electrodes and one set from a new electrode sheet. One set of the used discs was run with the matrix side from the life test toward the matrix and one set was run with the gas side from the life test toward the matrix. Compared with the performance of the unused electrodes, a much smaller loss is indicated in the latter run than the former. This would indicate that at least part of the voltage loss during the life test is dependent upon the nature of the matrix material used, and upon the extent that it penetrates the electrode(s) as the time increases.

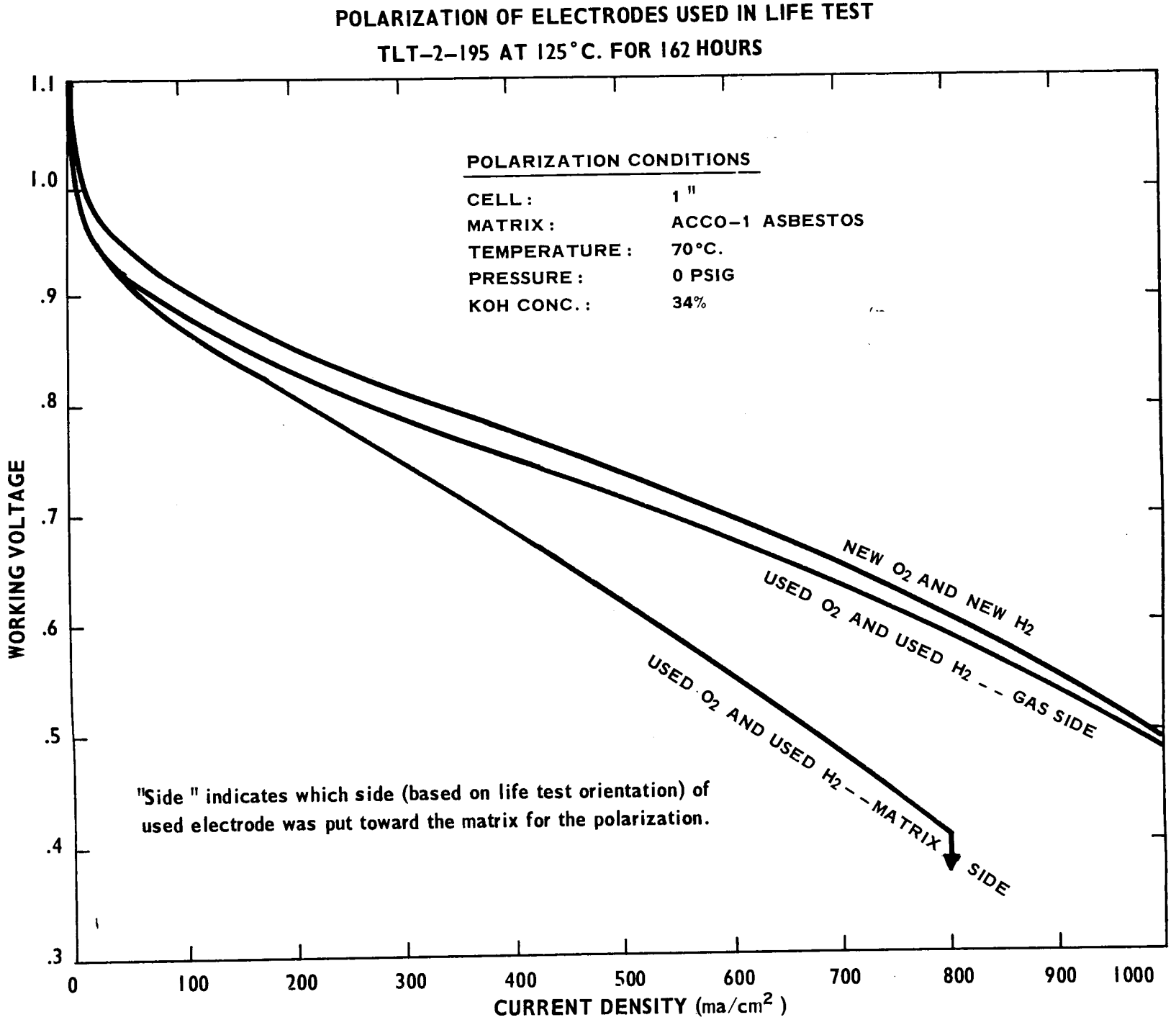
POLARIZATION OF ELECTRODES USED IN LIFE TEST
TLT-2-195 AT 125°C. FOR 162 HOURS
POLARIZATION CONDITIONS
CELL: 1"
MATRIX: ACCO-1 ASBESTOS
TEMPERATURE: 70°C.
PRESSURE: 0 PSIG
KOH CONC.: 34%
NEW O2 AND NEW H2
USED O2 AND USED H2 - - GAS SIDE
USED O2 AND USED H2 - - MATRIX SIDE
"Side" indicates which side (based on life test orientation) of used electrode was put toward the matrix for the polarization.
WORKING VOLTAGE
1.1
1.0
.9
.8
.7
.6
.5
.4
.3
0
100
200
300
400
500
600
700
800
900
1000
CURRENT DENSITY (ma/cm2)

FIGURE 3-6

4. FUTURE WORK

Work for the next quarter is planned for the following tasks.

1. Investigation of Operating Variables

Polarization tests will be run for the experimental design at 100-150°C and 0-60 psig using ACCO-II Asbestos as the matrix.

2. Materials Investigation

Evaluation of matrix materials for operation at 150-200°C will continue on a limited scale. Emphasis will be placed on PTFE compositions incorporating wettable fillers such as ceria.

3. Small Cell Life Testing

Life tests will be conducted at 125-150°C, atmospheric pressure and current densities of 100-400 ma/cm^2 using the following matrices (1) PTFE-asbestos (2) commercial porous PTFE (3) zirconia paper. Life tests will also be run at pressures above atmospheric using ACCO-II asbestos at 100°C.

4. Large Cell Fabrication

Single 6" x 6" cells suitable for operation up to 60 psig will be designed and fabricated.

5. Large Cell Testing

Preliminary life testing in 6" x 6" cells will be carried out at 100°C and atmospheric pressure over a range of current densities from 100-300 ma/cm^2.

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2. "Research and Development of High-Performance Light-Weight Fuel Cell Electrodes," American Cyanamid Company, First Quarterly Report, November 1, 1963, to January 31, 1964, NASA CR-54022.

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