FIRST QUARTERLY REPORT

CHARACTERIZATION OF RECOMBINATION AND CONTROL ELECTRODES FOR SPACECRAFT NICKEL-CADMIUM CELLS

by

W. N. Carson, Jr., G. Rampel and I. B. Weinstock

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

October 1966

CONTRACT NAS 5-10261

Goddard Space Flight Center
Greenbelt, Maryland

GENERAL ELECTRIC

Battery Business Section
Gainesville, Florida

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1.0 INTRODUCTION AND SUMMARY

1.1 Task I - Oxygen Sensing Electrode

The objective of this task is to develop an oxygen sensing electrode that will give a linear (or a reasonably close to linear response) to oxygen partial pressure in the range of approximately 5 to 30 psia. The initial approach being investigated is the use of diffusion controlled electrode reaction rates.

During the first quarter the following phases of this task were accomplished:

1. Oxygen sensing electrodes were fabricated with two film thickness (1/2 and 1 mil) of Teflon heat sealed to representative metallic porous and mesh type substrates.
2. Test cells to evaluate these sensing electrodes as a function of oxygen pressure, were designed and fabricated.
3. Test equipment to evaluate the electrodes was designed and fabricated. At the end of the quarter the equipment was assembled and being checked out in preparation for testing of electrodes.

1.2 Task II - Oxygen Recombination Electrode

The objective of this task is to select an oxygen recombination electrode capable of recombining the oxygen generated by the positive plate during charging. This electrode must be able to function over the temperature range of -20°C to +40°C. Selection of electrodes will be based upon their polarization characteristics.

During the first quarter the following phases of this task were accomplished:

1. Test cells for the characterization of electrodes were designed and built.
2. Electrodes of various structures and compositions have been fabricated and evaluated over the operating range.
3. The capability of recombination electrodes to perform over the entire -20°C to +40°C temperature range has been demonstrated.

1.3 Task III - Negative Plate Evaluation

The objective of this task is to select suitable lots of negative plate for use in prototype and final cell assembly. Lots will be selected upon their recombination ability and ampere-hour stability.

During the first quarter, two lots of plate were received from manufacturing and were subjected to standard aerospace formation cycles. Test cells incorporating these plates are being assembled.

2.0 TECHNICAL DISCUSSION

2.1 Task I - Oxygen Sensing Electrode

2.1.1 Experimental Approach

The initial approach is to use a diffusion-limited electrode coupled to a cadmium electrode. The current in such a cell depends upon the rate of duffusion of oxygen to the electrode surface, provided the electrode is held at a potential at which oxygen is readily and immediately reduced. Under these conditions, the diffusion rate of oxygen and therefore the current flow is directly proportional to the oxygen pressure in the gas phase. The problem then is to adjust the film thickness for a material having a given permeability for oxygen to give a linear response over the desired oxygen pressure range. Our initial approach is to evaluate the use of thin Teflon films which are heat sealed to various types of electrode substrate materials and forms.

2.1.2 Electrode Fabrication

The electrodes are fabricated by placing a film of Teflon (Dielectrix Co. Skivved sheet) on the substrate, covering the film with a smooth aluminum sheet, and pressing at a controlled pressure and temperature. The aluminum sheet is used to prevent sticking of the Teflon to the die faces. After pressing, the aluminum is removed by dissolving it in caustic.

The test electrodes fabricated to date and conditions of fabrication are listed in Table 1.

Table 1

DIFFUSION TYPE TEST ELECTRODES

Nickel Screen	1,	1/2,	1/4
Gold Mesh	1,	1/2,	1/4
Nickel Porous Sinter	1,	1/2,	1/4
Tantalum Screen, platinum coated	1	-	-
Tantalum Expanded Mesh, platinum coated	1	-	-

<u>Notes</u> - Pressing Conditions

1 mil films - pressure - 6 tons/in^2, temperature - 672°F, time - 45 minutes.

1/2 mil films - pressure - 4 tons/in^2, temperature - 672°F, time - 60 minutes.

1/4 mil films - to be done, conditions to be established.

2.1.3 Test Cells

The diffusion electrodes will be tested for polarization characteristics versus a cadmium counter electrode as a function of oxygen pressure. Two electrode sizes will be tested; one approximately 10 cm^2 in area and the other approximately 1 cm^2 in area. The components for the test cell are shown in Figure 1. The two electrodes are mounted on a Lucite sheet holder by means of an epozy cement. The holder is backed by a nylon separator, followed by the cadmium counter electrode, another piece of separator and finally a nickel screen counter electrode. The nickel electrode will be used to periodically recharge the cadmium electrode. The cell is fitted with a valve and manifold connector through which oxygen at a controlled pressure is delivered from a generator cell. The manifold and valve are also used to evacuate the cell of air prior to the start of the oxygen polarization tests. The test cell is encased in an outer cell. The space between the two cells is filled with a plastic to provide for pressure containment. This construction is shown in Figure 2.

2.1.4 Test Equipment

The oxygen consumed by the test cells is supplied by an electrolytic oxygen generator cell and a control system designed to maintain the pressure at a preset value. A schematic diagram of this system is shown in Figure 3.
The operation of this system is as follows. The test cells, generator cell and manometer detector are first evacuated and purged with oxygen. The system is then pressurized to

the desired operating pressure with oxygen. As oxygen is consumed, the mercury in the manometer surge chamber leg drops. When the mercury level falls below the lower of the two electrode probes the controller connects power from the constant current power supply to nickel and cadmium electrodes in the generator cell with oxygen being evolved from the nickel electrode. The rate of oxygen evolution is controlled by the current from the power supply. As the oxygen pressure builds up, the mercury level rises into the surge chamber leg, when it reaches the lower probe electrode power is removed from the nickel and cadmium electrode. In the event of a pressure overshoot, when the mercury level reaches the upper probe electrode, the controller connects the auxiliary electrode and the cadmium electrode to the constant current power supply. In this mode oxygen is consumed in the generator cell to reduce the pressure to the desired value. When the mercury level is in between the two probes, the generator cell is passive.

The electrical schematic and parts list for the oxygen pressure controller is shown in Figure 4. The completed controller along with the power supply for the oxygen generator is shown in Figure 5. The potted manometer detector and oxygen generator cell are shown in Figure 6. The system has been assembled and at the end of the quarter was being checked out.

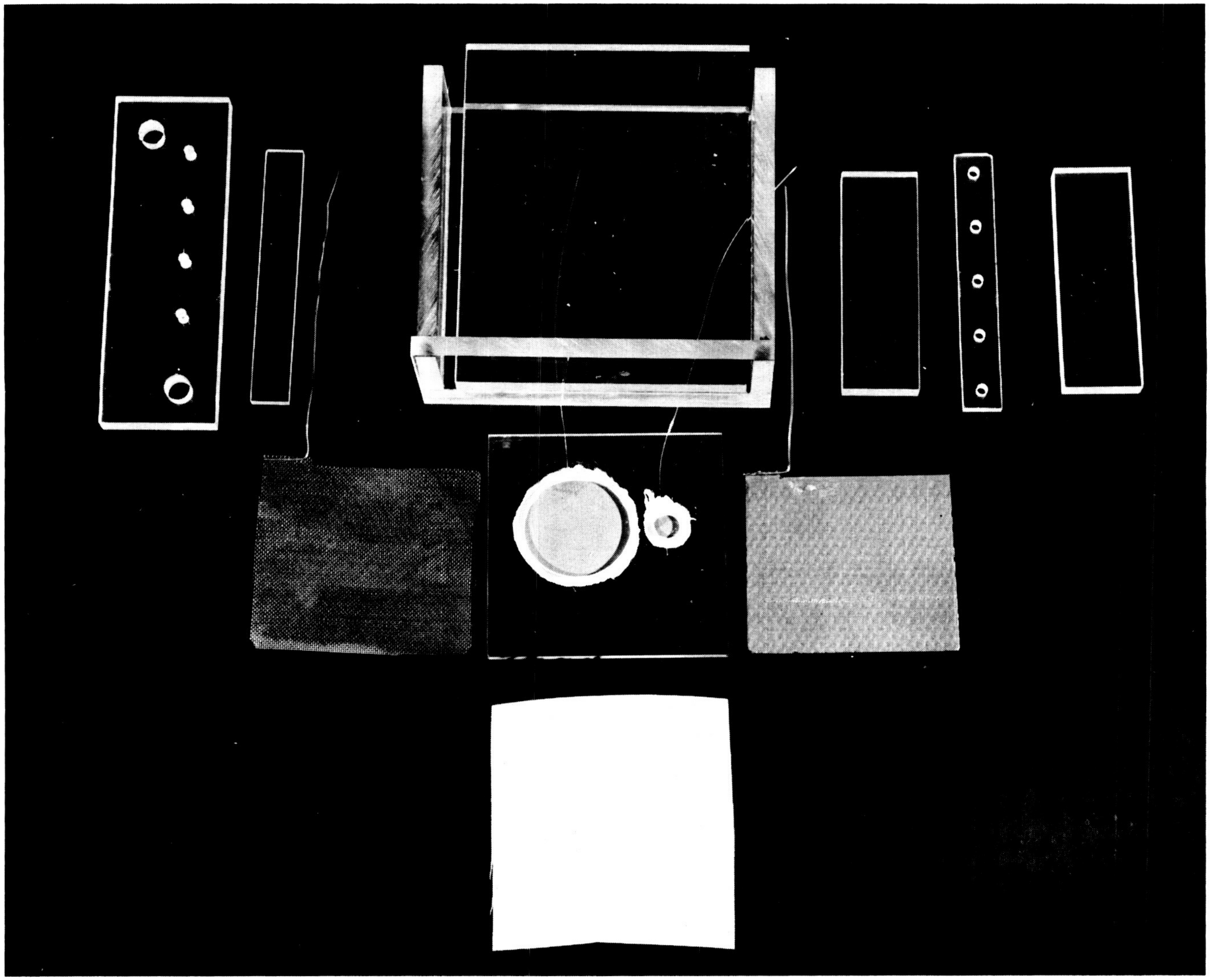

Figure 1

Oxygen Sensing Electrode Test Cell Components

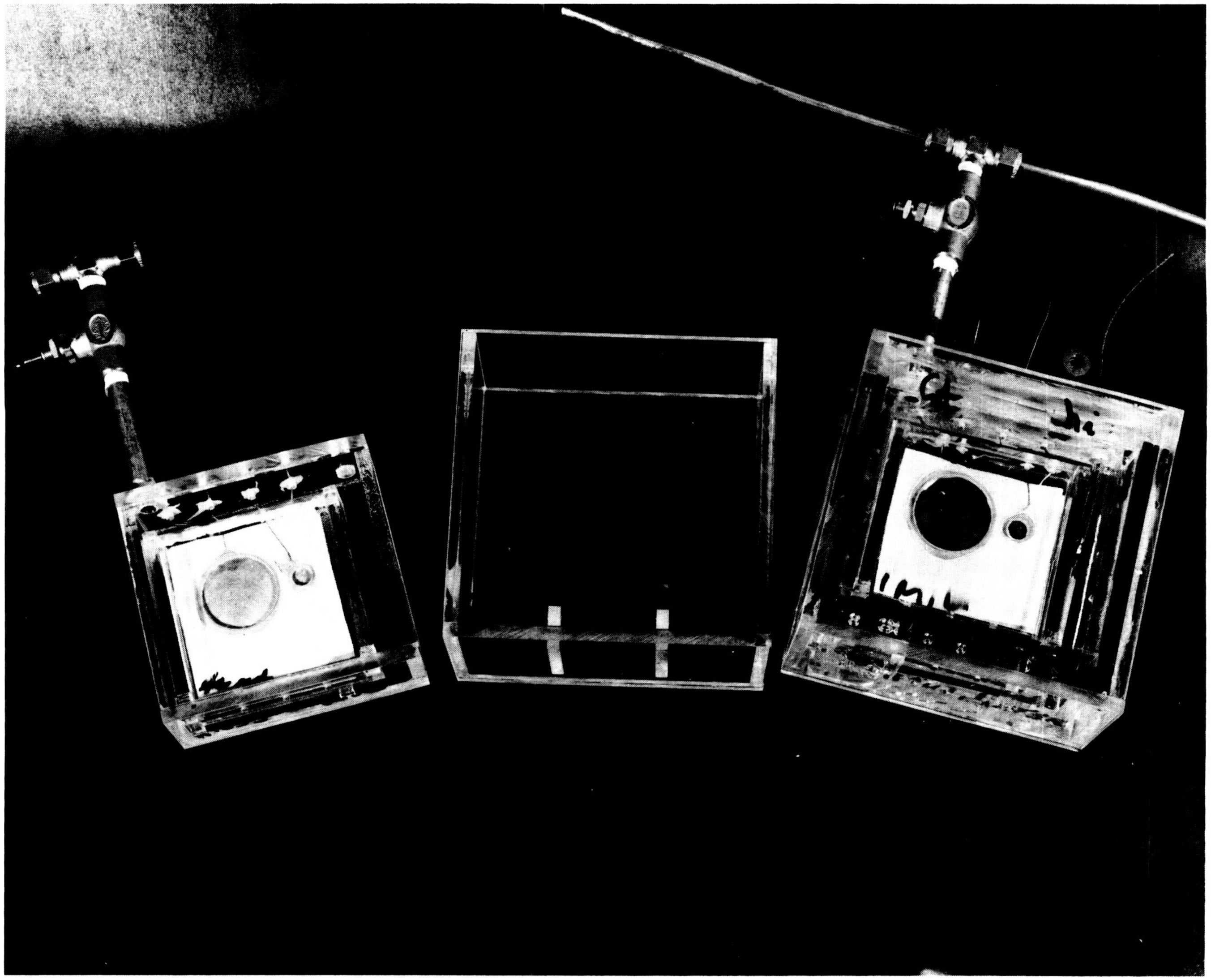

Figure 2

Oxygen Sensing Electrode Test Cell Assembly

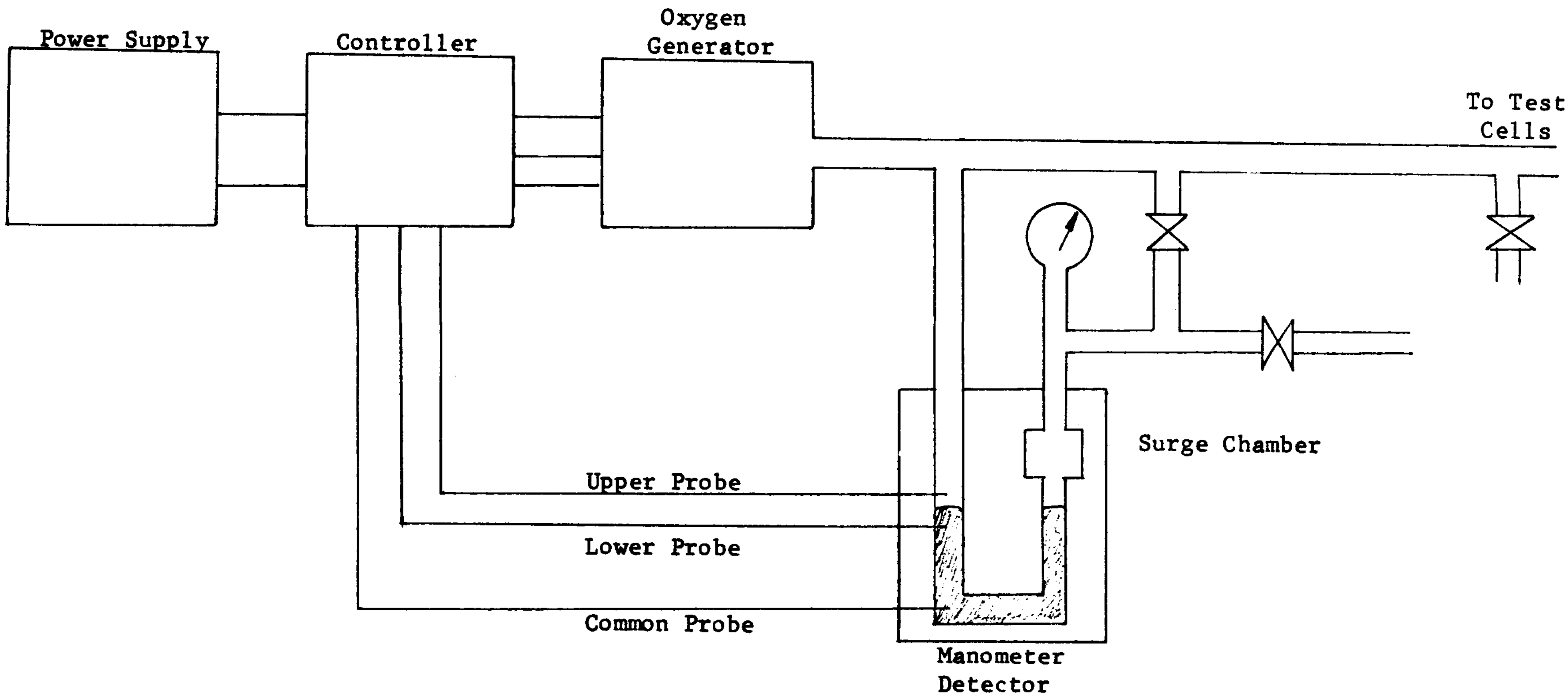

FIGURE 3.

Oxygen Electrode Testing System

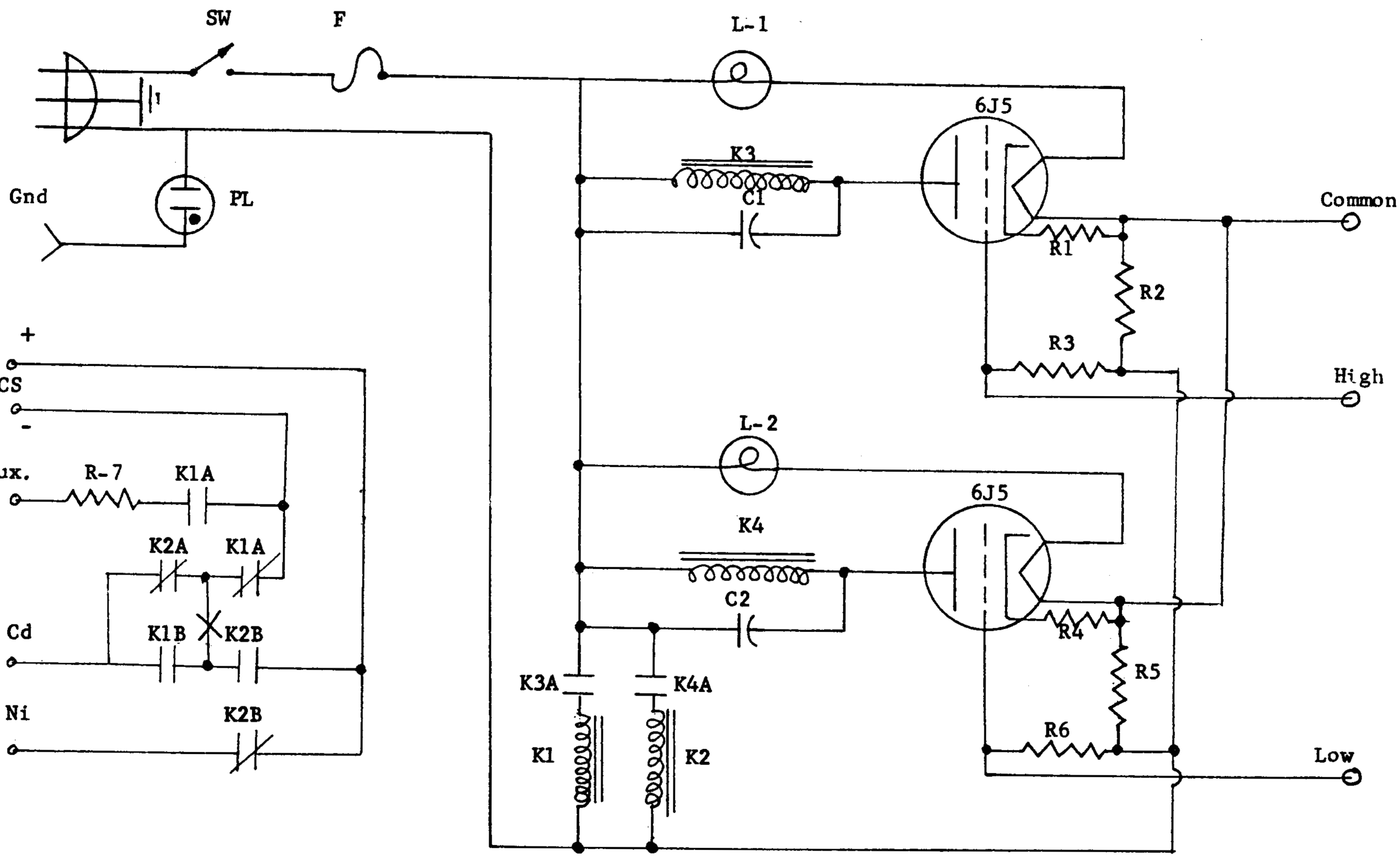

FIGURE 4

Electrical Schematic of Controller

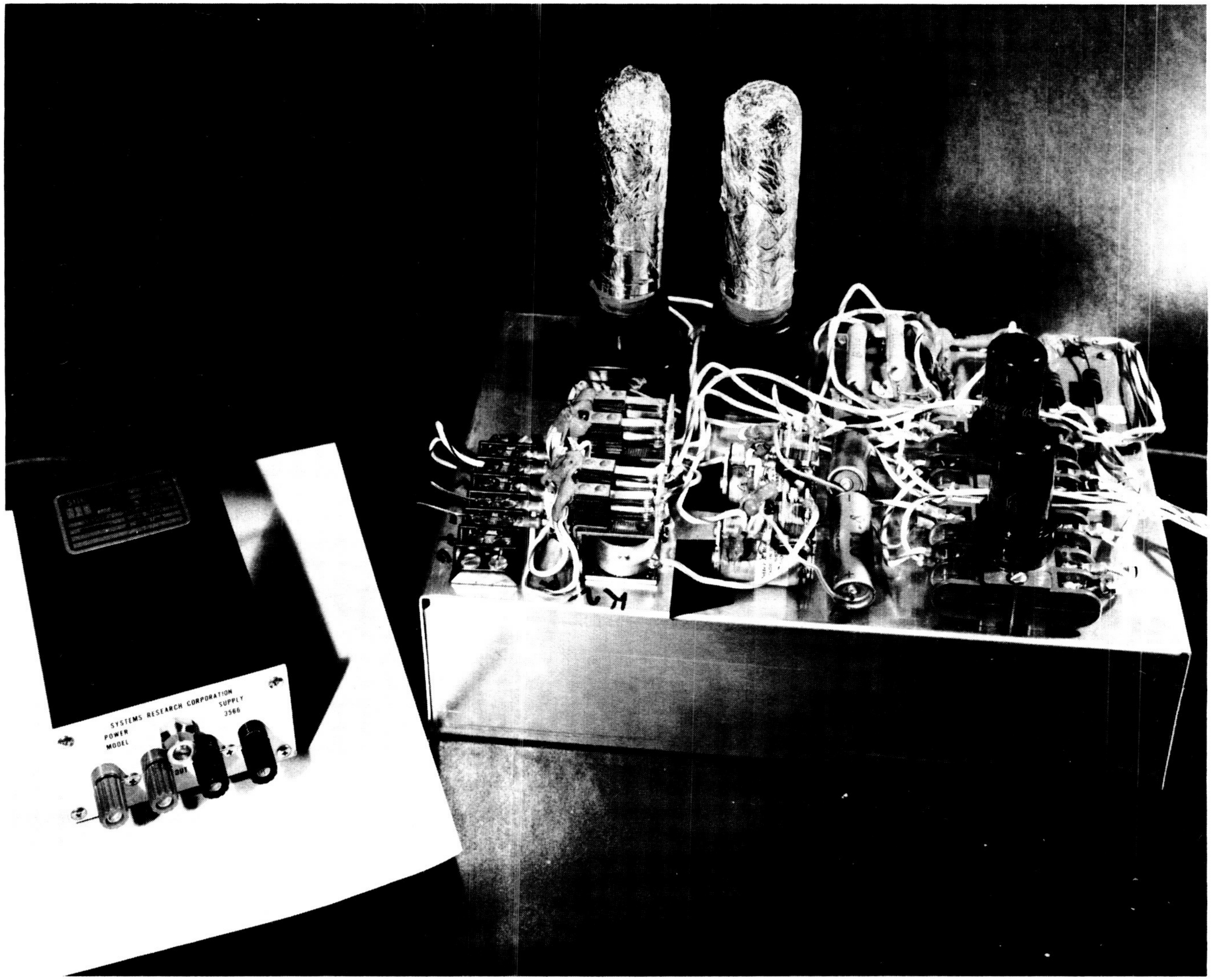

Figure 5

Photograph - Power Supply and Controller

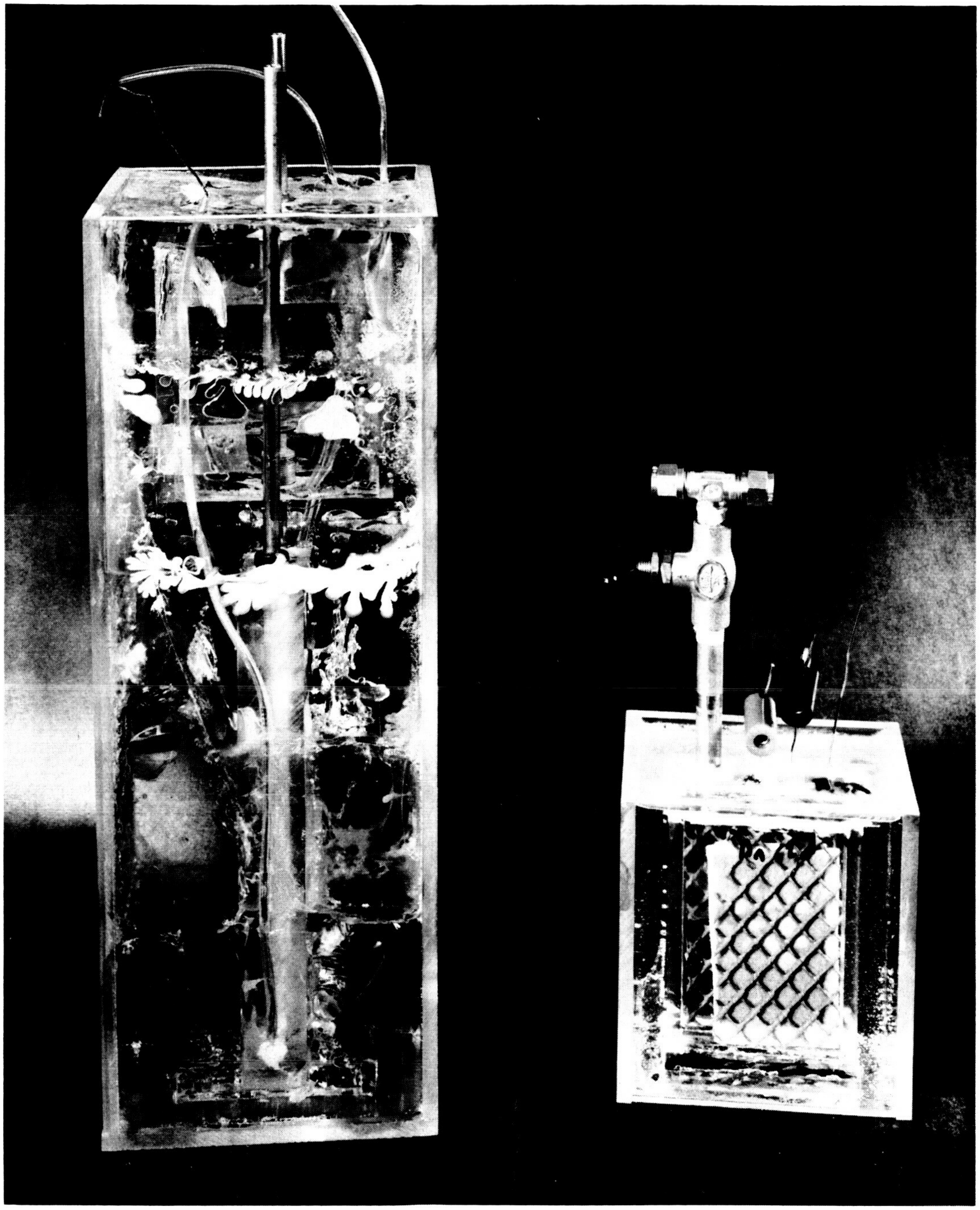

Figure 6

Photograph - Manometer Detector and Oxygen Generator Cell

2.2 Task II - Oxygen Recombination Electrode

2.2.1 Experimental Details

Recombination electrodes were evaluated by obtaining current density - voltage (polarization) curves as a function of operating temperature and oxygen pressure in cells as illustrated in Figure 7.

The test, counter, and reference electrodes are contained in a stainless steel can with a removeable cover. The cover is fitted with provisions for evacuating and/or pressurizing the cell and with a pressure gauge. Terminals for electrical connection to the various electrodes are also provided in the cover.

The counter electrode consists of a piece of sintered nickel plaque. The reference electrode is a piece of partially charged positive plate. The separator is non-woven nylon. The electrolyte is 31% KOH. The separator dips into a pool of electrolyte in the bottom of the cell, thus insuring a uniform degree of saturation in all tests.

The electrode and separator are assembled between two lucite plates as shown in Figure 8. The corrugated, perforated PVC provides access of O_2 to the face of the recombination electrode. Uniform compression is assured by applying constant torque to the four nickel plated screws holding the assembly together.

For operation, the test cells, after final assembly and pressure test, are evacuated to 30 inches Hg vacuum and back filled with O_2 to the desired pressure. They are then equilibriated for 16 hours at the test temperature. Following

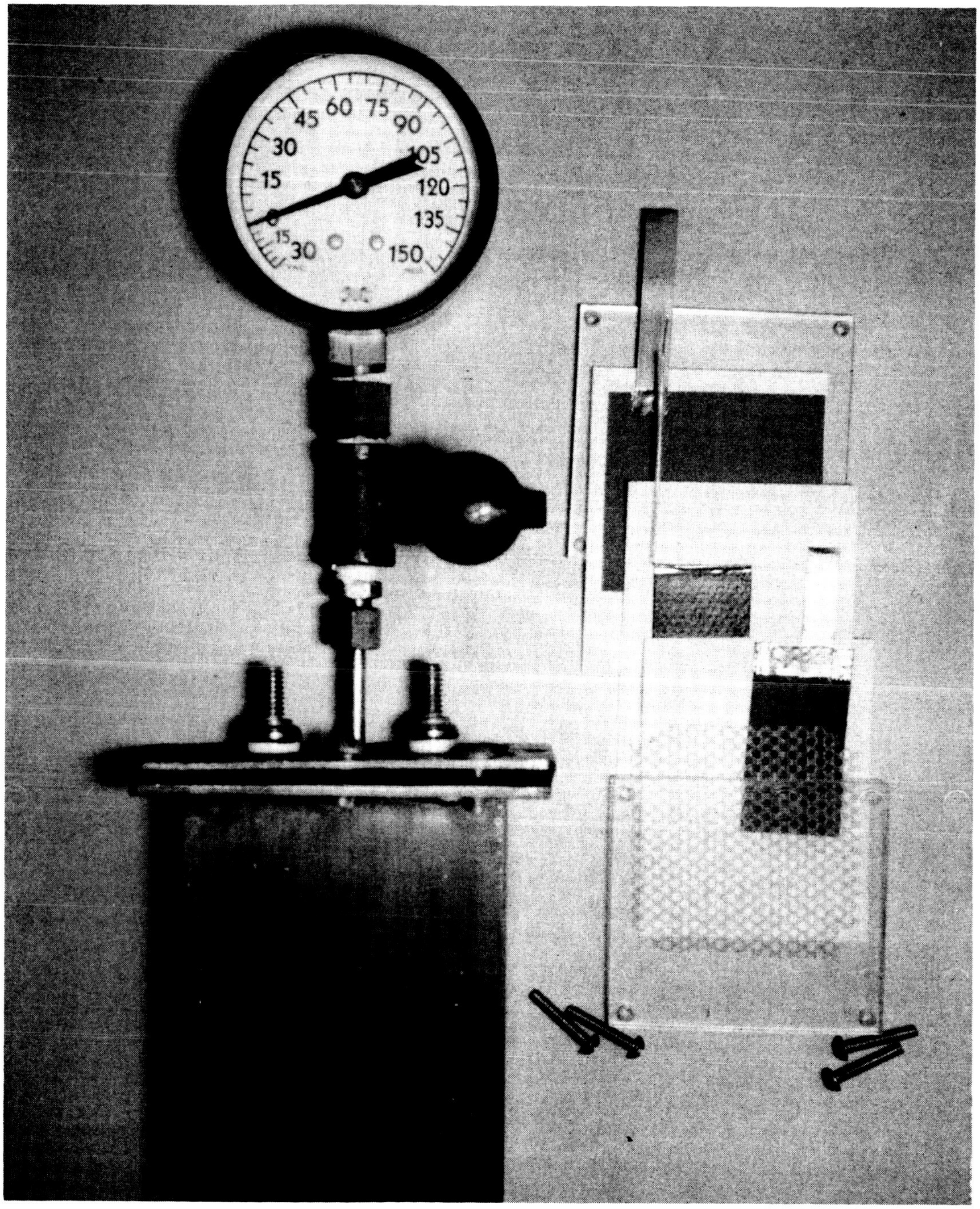

Figure 7

Recombination Electrode Test Cell
Components

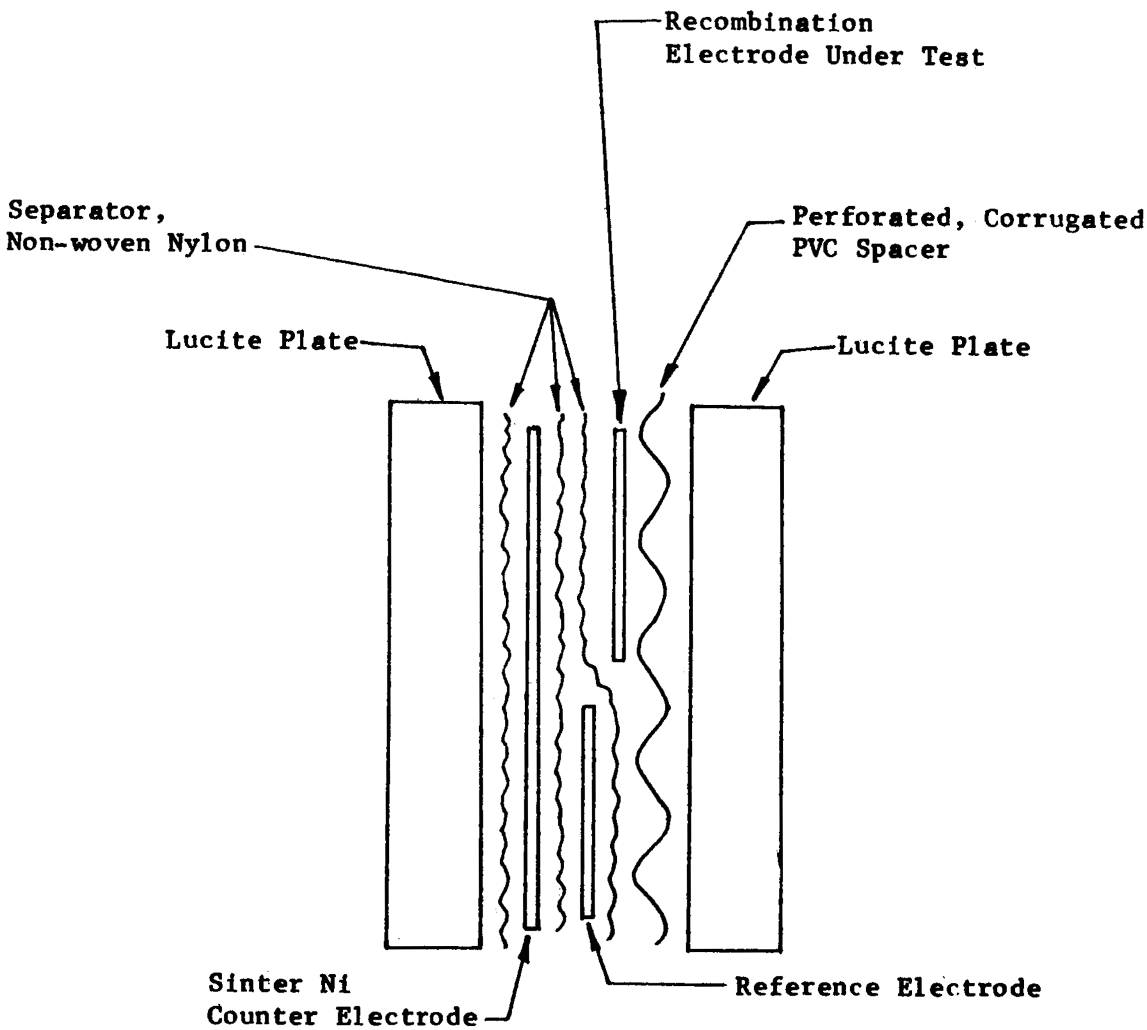

FIGURE 8

Electrode Assembly for
Recombination Electrode Evaluation

this temperature stabilization, current is passed between the counter and test electrodes, causing O_2 to be generated on the former and recombined on the latter. The voltage between the test and reference electrode is measured at each current level. Hydrogen evolution on the test electrode, if allowed to occur, can be observed by a rise in the cell pressure and also by the test vs reference electrode voltage.

2.2.2 Electrode Fabrication

Recombination electrodes are fabricated by application of Pt catalyst to a substrate consisting of nickel powder sintered to an expanded nickel strip. The electrodes are wet-proofed by application of a Teflon suspension to the gas face of the electrode and subsequently curing at elevated temperature. Details of the electrodes fabricated during this period are presented in Table II.

2.2.3 Results and Discussion

The performance of the first group of electrodes evaluated is presented in Figure 9. As can be seen, the electrodes are capable of recombining oxygen at substantial rates at moderate temperatures and pressures, but that they function poorly at the extremely low temperature. While a decrease in performance is to be expected with a decrease in operating temperature, the extent to which this occurs may be modified by altering the structure of the electrode. Such alterations would include plaque structure, catalyst loading, and wetproofing.

Such modifications were evaluated in the electrodes of groups 2 through 4. Typical results for various of these are presented in Figures 10 and 11. In Figure 10 are presented

Table II

DETAILS OF RECOMBINATION ELECTRODE FABRICATION

Group	Electrode Number	Sintered Ni Plaque Lot	Thickness mils	Porosity %	Catalyst mg/cm^2	Teflon mg/cm^2
1	183	5-C	10	74.5	1.23	1.96
	184	5-C	10	74.5	1.25	1.84
2	192	8	14	81.2	2.69	1.77
	193	8	14	81.2	2.59	1.24
	195	8	14	81.2	2.67	1.55
3	R-1	5	11	85.4	2.62	0.96
	R-2	5	11	85.4	2.70	1.23
	R-3	5	11	85.4	5.00	1.12
4	500-1	5	11	85.4	5.16	1.19
	500-2	5	11	85.4	5.08	1.17
	500-3	5	11	85.4	10.29	1.23

the polarization curves for electrodes R-3 and 193 at -25°C and 25 psia of O_2 pressure. It can be seen that the use of a thinner, more porous plaque results in an electrode with improved low temperature performance. The effect of catalyst loading is presented in Figure 11, and an increase in the performance is observed with an increase in catalyst loading.

2.3 Task III - Negative Plate Lot Evaluation

2.3.1 Work Done During Quarter

Two lots of negative plate were received from manufacturing for evaluation. A sufficient number of plates to construct 12 cells from each lot were cut to size and subjected to standard aerospace formation cycles. The plates were then washed free of electrolyte and dried. The plates are currently being assembled in sealed, semi-starved test cells for cycle testing in order to determine their recombination ability and ampere-hour stability.

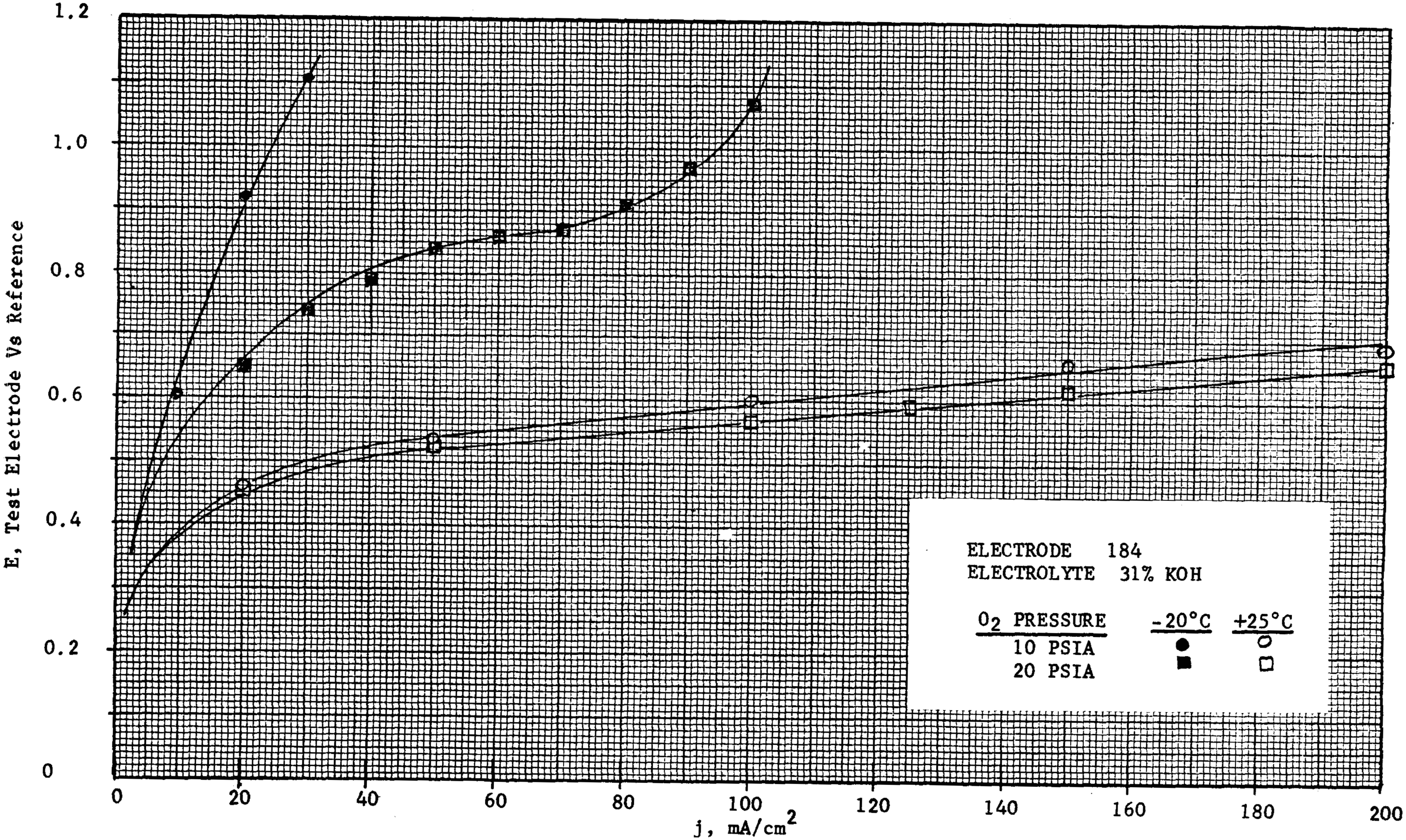

FIGURE 9: Performance of Recombination Electrode No. 184 at Various Temperatures and Pressures

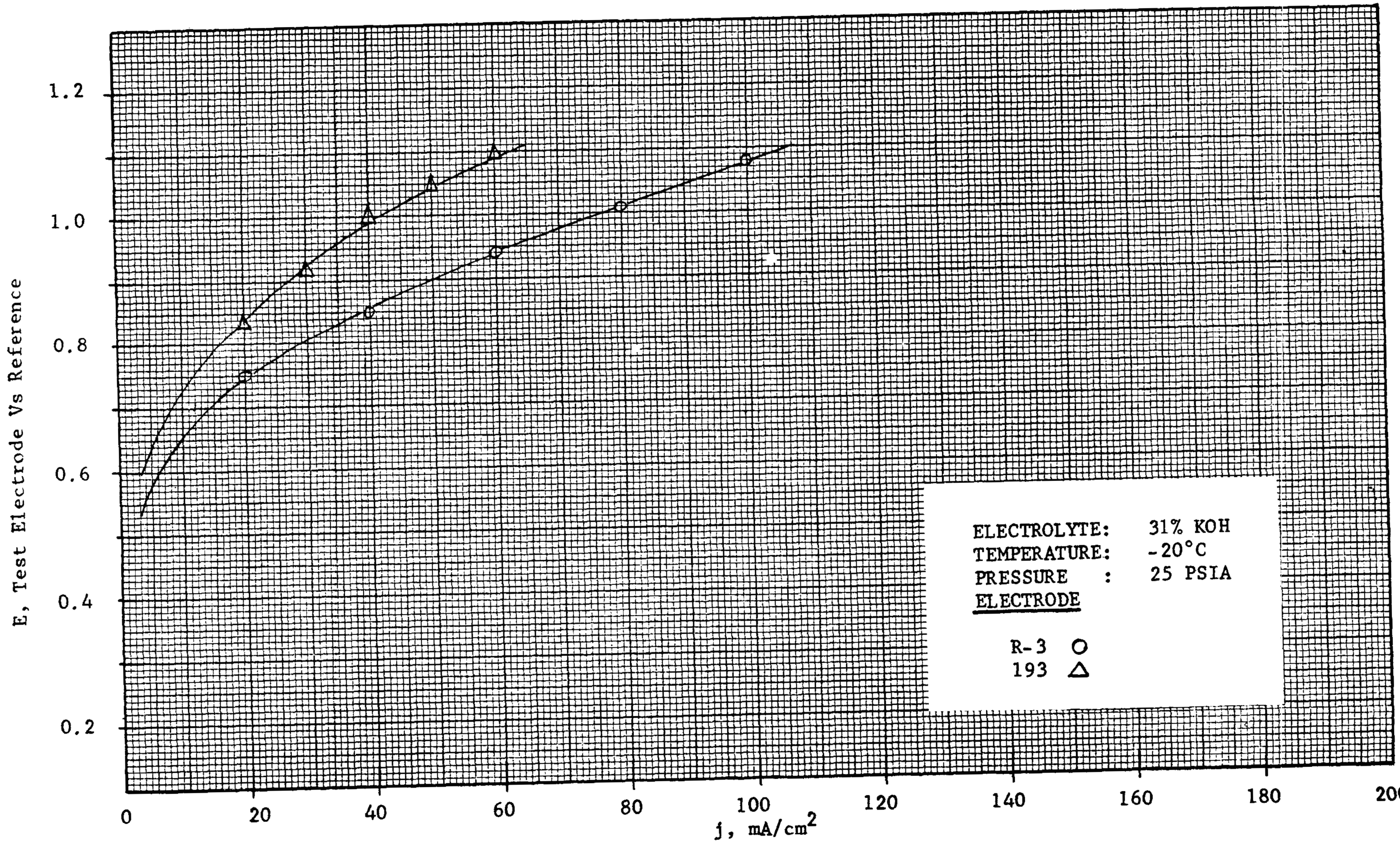

FIGURE 10: Effect of Plaque Structure on Low Temperature Performance

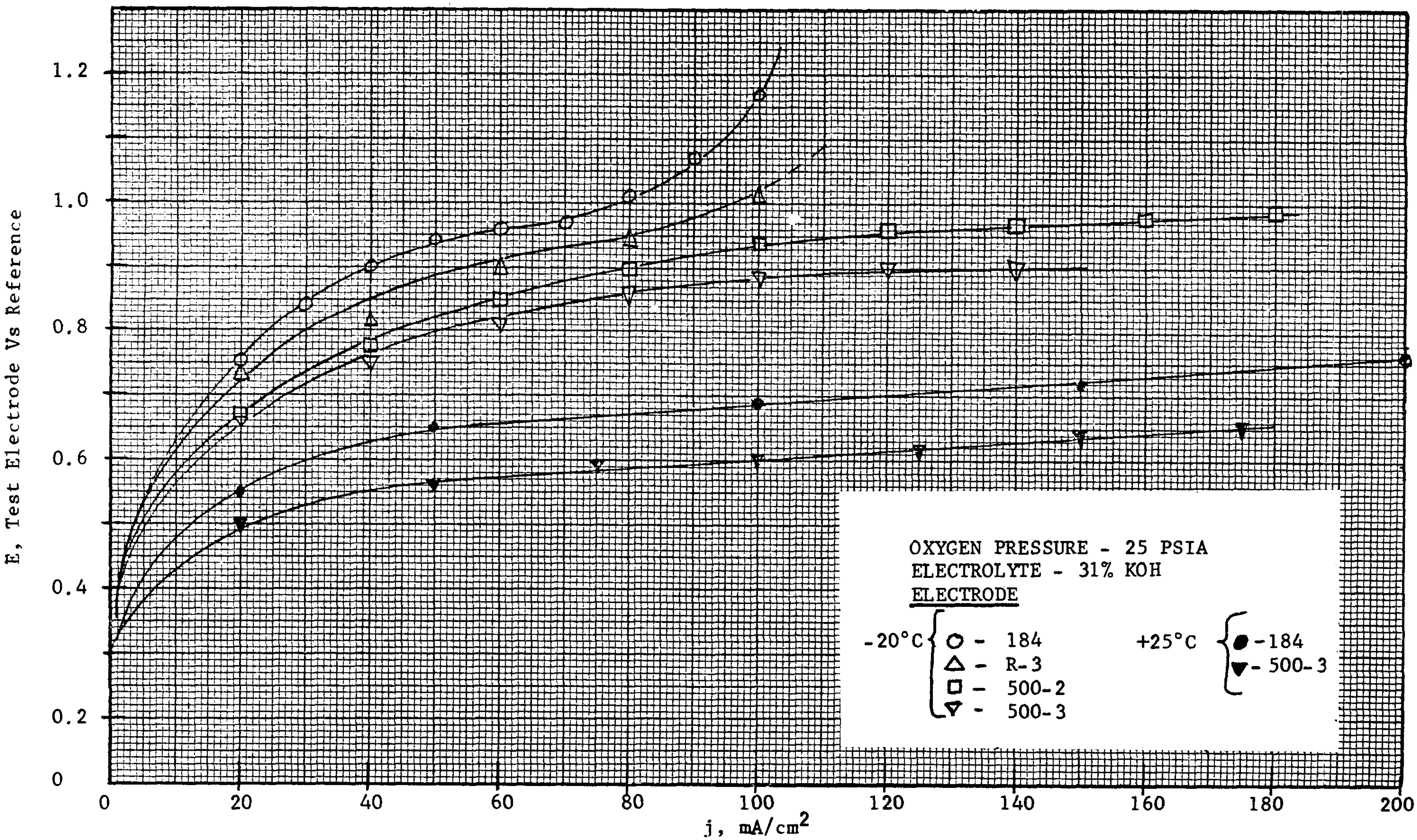

FIGURE 11: Effect of Catalyst Loading on Electrode Performance

3.0 WORK PLANNED FOR NEXT QUARTER

3.1 Task I - Oxygen Sensing Electrode

Additional electrodes will be fabricated as required. The electrodes now in hand will be tested for polarization characteristics over an oxygen pressure range of 5 to 30 psia and for temperature effects from -20°C to +40°C. The more promising candidate electrodes will be tested for longer term behavior, reproducibility, and internal cell impedance effects.

3.2 Task II - Oxygen Recombination Electrode

Additional electrodes will be fabricated in order to confirm the results presented above, and to extend the range of the variables. Work directed at determining the reproducibility of the electrodes has been initiated.

A change in the design of the test cells is also being made. This change will allow for more uniform heat transfer from the working electrodes to the atmosphere. Provision for temperature measurement within the cell will also be made. These changes will allow for more uniform testing.

3.3 Task III - Negative Plate Lot Evaluation

One additional lot of negative plate will be obtained from manufacturing for evaluation. One lot from the three evaluated will be selected, on the basis of recombination ability and ampere-hour stability, for use in prototype and final cell assemblies.

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