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ADVANCED FUEL CELL CATALYSTS
AUGUST 10 to NOVEMBER 10, 1969

**Pennsylvania
Research
Associates, Inc.**

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PENNSYLVANIA RESEARCH ASSOCIATES INC.
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STATUS REPORT

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ADVANCED FUEL CELL CATALYSTS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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TABLE OF CONTENTS

	<u>PAGE</u>
A. Abstract, Conclusions, and Recommendations	1
B. Factual Data	3
B.1 Catalyst Preparation	3
B.1.a Melting	4
B.1.b Catalyst Grinding	5
B.2 Size Changes and Powder Color	8
B.3 Electrodes	10
B.3.a Electrode Fabrication	10
B.3.b Experimental Results	12
B.3.c PRA Electrodes as Anodes	19
B.4 Catalytic Performance as a Function of Particle Size	22
B.4.a Size Variation	22
B.4.b Separation of Fines	24
B.5 Life Test Fuel Cells	27
C. Discussion	29
C.1 Improved Catalyst Chemical Resistance	30
C.2 Improved Electrode Resistance	31
D. Plans for Next Quarter	32

TABLE OF FIGURES

<u>FIG #</u>		<u>PAGE</u>
1	-- Ground catalyst x 1,000; scale in microns	9
2	-- Performance of early black with porous teflon	13
3	-- H ₂ -O ₂ fuel cell electrode measurements: variations in AB-40 performance in oxygen reduction	14
4	-- H ₂ -O ₂ fuel cell catalyst investigation; comparison of platinum electrodes for oxygen reduction	16
5	-- Comparison of PRA Pt electrode performance as cathode and anode	17
6	-- H ₂ -O ₂ Fuel cell catalyst investigation; PRA electrode comparison Pt vs. Ag	18
7	-- Performance status of PRA catalysts for oxygen reduction	20
8	-- Comparison of PRA catalysts as anodes and cathodes in H ₂ -O ₂ fuel cells	21

A. Abstract, Conclusions, and Recommendations

During the past quarter emphasis was on preparing large batches of catalyst. A 20-kw induction furnace replaced a 3-kw resistance furnace; as a result the production of catalyst from the shot tower increased from a few grams to 3 kilograms per week.

The single-crystal, $\sim 250\mu$ balls are first ground in an aluminum oxide mortar (Fisher Scientific Co.) under glycerol for 72 hours at room temperature in air, then further ground in an attritor ball mill (Union Process Co., Akron, Ohio) with glycerol 80% and distilled water 20% for 72 hours at room temperature in air. Using a calibrated reticle in the optical microscope we estimate the final particle diameter at $.5\mu$ and less. We estimate the output after grinding to be about 150 grams per 40-hour week.

The new method was used to prepare ~ 250 grams of catalyst, samples including eleven gold-copper alloys (pure or doped with 10, 15, or 22 at-% In, Ga, or Al) and three silver powders (one pure, two doped with 10 at-% Al). Depending on their average size, the powders varied in color from light tan ($\sim 10\mu$) to blacks ($\lesssim .5\mu$).

Electrodes constructed with all but the black powders were inferior in performance as compared with our earlier preparations. A simple analysis (see Section C) has shown the importance of removing coarse particles, if maximum activity is to be obtained. We are,

therefore, building an air floatation device for collecting and separating fines into groups, for instance, in three groups, one consisting of particles less than 0.5 μ in diameter, another one of particles smaller than 3 μ but larger than 0.5 μ , and the third of the largest particles.

Three life-test stations should become available by December 16, 1969.

B. Factual Data

B.1 Catalyst Preparation

During the past quarter the work re-oriented because of the need for producing considerably larger quantities of more uniform, finer powders than was possible before. We desired, as an initial goal, to be able to produce up to 100 g per week of powder of less than 0.5μ in diameter.

This required two changes:

(1) a more powerful metallurgical furnace, which consists of an induction R.F. heater of 20-kw output. This replaced a 3-kw resistance heater. The new furnace can produce two to three kilograms a week of the small, ($\sim 250\mu$) uniformly sized, single-crystal balls we use as starting material for grinding.

(2) a different grinding technique as the original output was limited to about two grams a week. We needed much more material just for testing ways of making electrodes.

B.1.a Melting

Catalyst materials during this period were prepared with an induction furnace installed in mid-September 1969. This unit has allowed a large improvement in efficiency of preparation, in terms both of time, reduced from about $4\frac{1}{2}$ hours to 30 minutes per run, and uniformity of temperature, the original furnace causing a variation of as much as 16°C between the ends and the center of the crucible. With the new furnace the variation cannot be read using an optical pyrometer (Leeds & Northrup cat. 13-877-10, cent. 775° to 2000°).

The induction furnace is a water-cooled 20-kw - 220 v single-phase continuous-input Ajax Northrup High Frequency Converter which makes use of an H_2 -pressurized spark gap. This unit has been adapted to the Material Research Corporation's vertically mounted bed previously used in our "shot tower" technique. The induction coil can thus be properly positioned with respect to the carbon crucibles in which materials are melted. Melting times are now a matter of 1-5 minutes for our crucible dimensions ($1\frac{3}{4}$ " i.d. X 8") rather than several hours. To date the highest temperature attained is 1525°C in one crucible as determined with a Leeds & Northrup optical pyrometer.

Our technique is to melt the material 3-5 times inside the crucible, each time allowing solidification and then reversing the resulting metal cylinder within the crucible. Small holes (about 0.006" diameter) in the base of the crucible allow the melt to be forced

out under pressure (up to 40 psig of argon) into a slightly pressurized (2-5 psig) nitrogen atmosphere quartz shot tower of 2" i.d. and about 8' height. Very small spherical balls are collected at the base; their sizes vary from a few tens of microns to several hundreds. Distributions of ball sizes vary, of course, with the size, number, and irregularity of drilling of the holes in the crucible.

The shorter catalyst melting times now have resulted in a backlog of materials awaiting the relatively limited grinding facilities on hand (see p.11).

B.1.b Catalyst Grinding

Originally, the crystalline materials were ground in an aluminum oxide mortar from Fisher Scientific Company. This resulted in a fairly coarse powder. This powder, when further ground in tungsten carbide mortars (Fisher Scientific Company), produced a satisfactory product. But the tungsten carbide mortars are small and hold only 2-3 grams of powder. In both mortars Triton-X-100 (Rohm & Haas) and ethyl alcohol (A. H. Thomas) were used as grinding media. The ethyl alcohol evaporated so that unattended operation overnight was impossible.

About the beginning of the quarter we substituted glycerol as a grinding medium. It does not evaporate and appeared satisfactory, as the fines resulting from prolonged grinding were at least as small as those previously obtained. However, the initial results

of grinding in glycerol were tan or brown powders, whereas previous grinding always produced black powders. The implications of these color changes were not immediately understood.

The output would still have been very small, even using glycerol. Hence, we attempted to utilize an attritor ball mill (Union Process Inc., Akron, Ohio) for producing fines. These mills take as least 100 grams at a time.

The mills are new, and their proper use is a matter of considerable trial and error. After having produced mediocre materials for a relatively long time, they now yield the finest and most uniform material we have obtained.

Two grinding techniques are being used. One is a mechanized mortar-pestle of rather small volume, and the other is an accelerated ball mill operation. The former uses Fisher Mortar Grinders 8-323 V (2) and the latter an electrically stirred Attritor Ball Mill, type B, size 01 from Union Process Inc. of Akron, Ohio. The material of the Fisher mortar is alumina; the attritor balls are 3/8" stainless steel.

Originally grinding was done in denatured ethyl alcohol to which a trace of Triton X-100 (Rohm & Haas) surfactant was added. Evaporation of the alcohol prevented continuous grinding, and also resulted in some of the fines escaping from the mortar during grinding.

We have since changed to glycerol (Matheson, Coleman & Bell, GX M5) in the mortar and an 80-20 % glycerol-water solution for holding the materials in suspension in the attritor. Continuous grinding is carried out without significant loss of material.

Our original fine grinding operation was undertaken with a tungsten carbide mortar-pestle combination of limited capacity, roughly 1/10 that of the alumina combination. While very fine grinding of our materials is attainable in these units, our output was limited to 2-3 grams of powder per week.

Originally the attritor was used unsuccessfully with large diameter particles of the relatively soft material AuCu plus 15 at-% Al. No fine powder resulted, but the balls were merely flattened into comparatively large platelets.

Subsequently, we learned that the attritor requires much smaller initial particle sizes (100 mesh) which we can attain with 96 hours alumina mortar grinding. This resulted in much improved attritor grinding, and it is now being investigated in terms of time and stirring speed. We find color changes in the fines going from tan to brown to a deep black as our average particle size becomes smaller and approaches that of visible light wavelengths. Inspection with an optical microscope of magnifying power up to 2,000 shows the decreased sizes as attritor grinding times are increased.

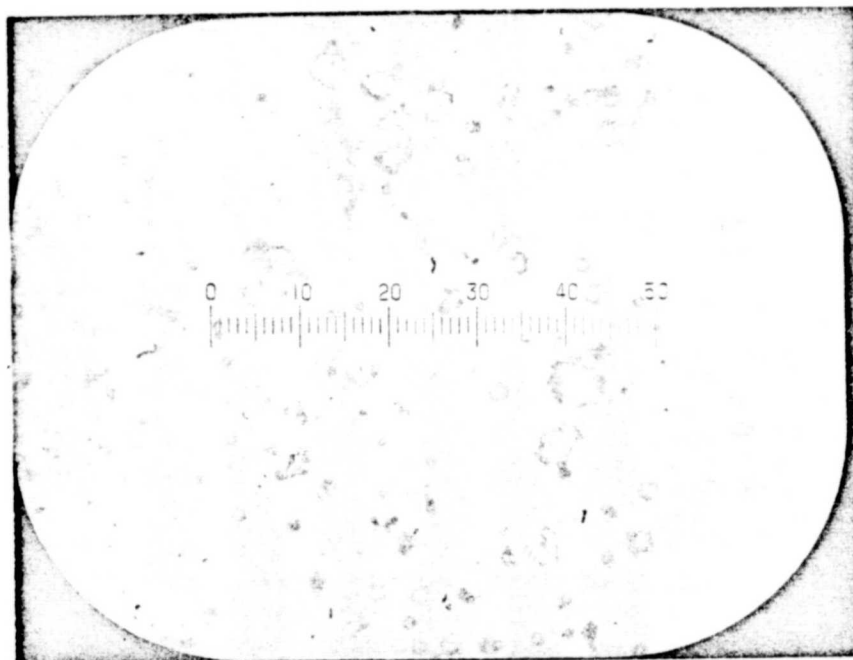
A comparison between a brown and a black powder as seen by the microscope with 1,000 magnification is shown in Fig. 1. The finished powders are repeatedly washed in alcohol and distilled water; the fines are precipitated out of suspension using a small centrifuge. Centrifuging is faster than letting the particles precipitate out of a water suspension. Nevertheless, centrifuging, particularly so for the finest powders, is slow, taking about a day for an 80-gram load. It has become the bottleneck of catalyst production. The powders when dry are ready for use in electrodes.

B.2 Size Changes and Powder Color

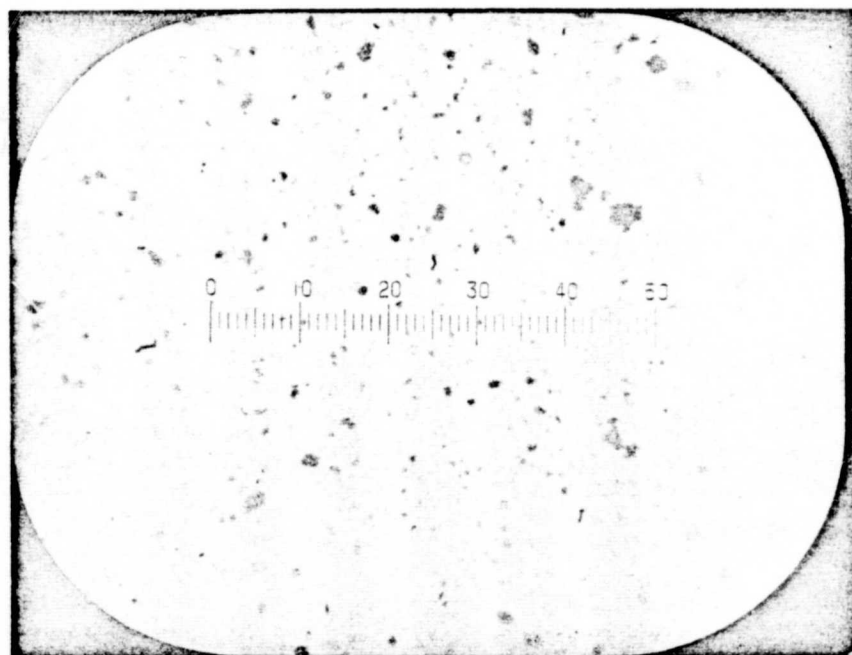
We have tried to correlate electrode performance to the color of the powders. The colors change with decreasing size and percentage of larger particles and range from tan to deep black.

At the same time as the color changes, the apparent density of the particles also changes: representative values are about 3.6 grams/cm³ for the tans, 2.3 for brown, 1.01 for some early blacks to a minimum of 0.73 g/cm³ for the latest products of the attritor.

For a tan colored powder all the particles are relatively large as observed in the optical microscope, ranging from 1 micron to 10. The spread of particle sizes is not uniform but may approximate a Maxwellian distribution. For a very dark brown the sizes appear to range



1a Brown - The larger spots are individual crystals and reflect light



1b Blacks - The larger spots are aggregates

from $\frac{1}{2}$ to 4 micron, again with a roughly Maxwellian distribution. For an early black (grey black) from a tungsten carbide mortar about half of the population may be $\frac{1}{2}$ micron or less. The rest are between 1 and 3 micron.

Finally a full black after 72 hours grinding in the attritor is nearly exclusively composed of particles $\frac{1}{2}$ micron or less in diameter. Perhaps one in 10^4 is larger, of the order of 1-2 micron.

B.3 Electrodes

B.3.a Electrode Fabrication

A list of ground materials as available toward the end of this period is as follows:

<u>Prep. #</u>	<u>Composition; at-%</u>	<u>Amount, Grams</u>	<u>Grinding Conditions</u>	<u>Color</u>
1	AuCu ₃	.35	glycerol	reddish brown
20	AuCu ₃ + 12 Al	14.21	4 days in glycerol	grey brown
4	AuCu ₃ + 15 Al	13.81	6 hrs in attritor - glycerol and water	greyish med. brown
3	AuCu ₃ + 15 In	10.03	glycerol	grey brown
21	AuCu ₃ + 15 Ga	13.88	glycerol	light grey brown
5	AuCu ₃ + 15 Ga	18.09	alumina mortar	yellow-grey
7	AuCu ₃ + 22 Al	5.47	triton X-100	brown black
9	AuCu ₃ + 22 Al	13.38	4 days, 8 hrs in attritor	blackish
6	AuCu ₃ + 22 Al	85	4 days, 100 hrs	black

<u>Prep. #</u>	<u>Composition; at-%</u>	<u>Amount, Grams</u>	<u>Grinding Conditions</u>	<u>Color</u>
8	AuCu + 10 Al	23.50	4 days in X-100	light brown
13	AuCu + 15 Al	7.22	glycerol	medium brown
11	AuCu + 15 Al	1.75	glycerol and X-100	light brown
22	AuCu + 15 Al	2.99	glycerol and X-100	medium grey brown
12	AuCu + 15 Ga	4.92	glycerol	light brown
19	AuCu + 22 Al	.71	X-100	black
10	AuCu + 22 Al	23.67	X-100 then glycerol	dark brown
23	AuCu + 22 Al	21.23	glycerol	light brown
14	AuCu + 22 Al	19.73	glycerol and water	greyish brown
2	Ag + 10 Al	4.26g	glycerol	light grey

Materials thus far prepared and being processed but not yet fully ground, are:

AuCu + 22 Al	10g	AuCu ₃ + 22 In	20g
AuCu + 22 In	10g	AuCu ₃ + 22 Ga	10g
AuCu + 22 Ga	10g	AuCu ₃ + 15 Al	45g
AuCu + 15 In	10g	AuCu ₃ + 15 In	10g
AuCu + 10 Al	15g	AuCu ₃ + 15 Ga	10g
		AuCu ₃ + 10 Al	14g
AuCu ₃	50g	AuCu ₃ + 10 In	15g
		Ag + 15 In	20g
		Ag + 10 Al	67g
		Ag	15g

B.3.b Experimental Results

The materials used during this period were browns (see Section B.3.a) of an average bulk density of 2.8 g/cm^3 [as compared with Pt black (0.5 to 1.3 g/cm^3) or with our recent alloy black (0.73 g/cm^3)]. The relatively coarse materials, averaging perhaps 3μ , have specific areas that we estimate to be around $1/10$ those of the latest blacks.

Performance was mediocre, with the exception of one electrode with a porous Teflon film and an early black which gave a good initial performance (see Fig. 2). This technique is being pursued.

Figs. 3-7 show results of short-term testing of our electrodes.

Fig. 3, data taken here and at Tyco* with AB-40 electrodes (American Cyanamid), shows the variability of this material when used as a cathode. The current density obtained with three samples of AB-40, at a given cell voltage, varied by more than 2 to 1 over a large portion of the range shown. Variation with time for a given piece of the material, as shown by the Tyco data, is significant at higher currents. The PRA data were taken under slightly different conditions from those at Tyco, but the differences in concentrations (5 vs. 8N KOH) and pressures (15 vs. 10 psig) tend to offset one another.

* "Development of Cathodic Electrocatalysts for Use in Low Temperature H_2/O_2 Fuel Cells with an Alkaline Electrolyte", Final Report covering the period from 1 July 1965 to 30 June 1968, Contract NASW-1233 Q-12, Tyco Laboratories, pp. 172-186.

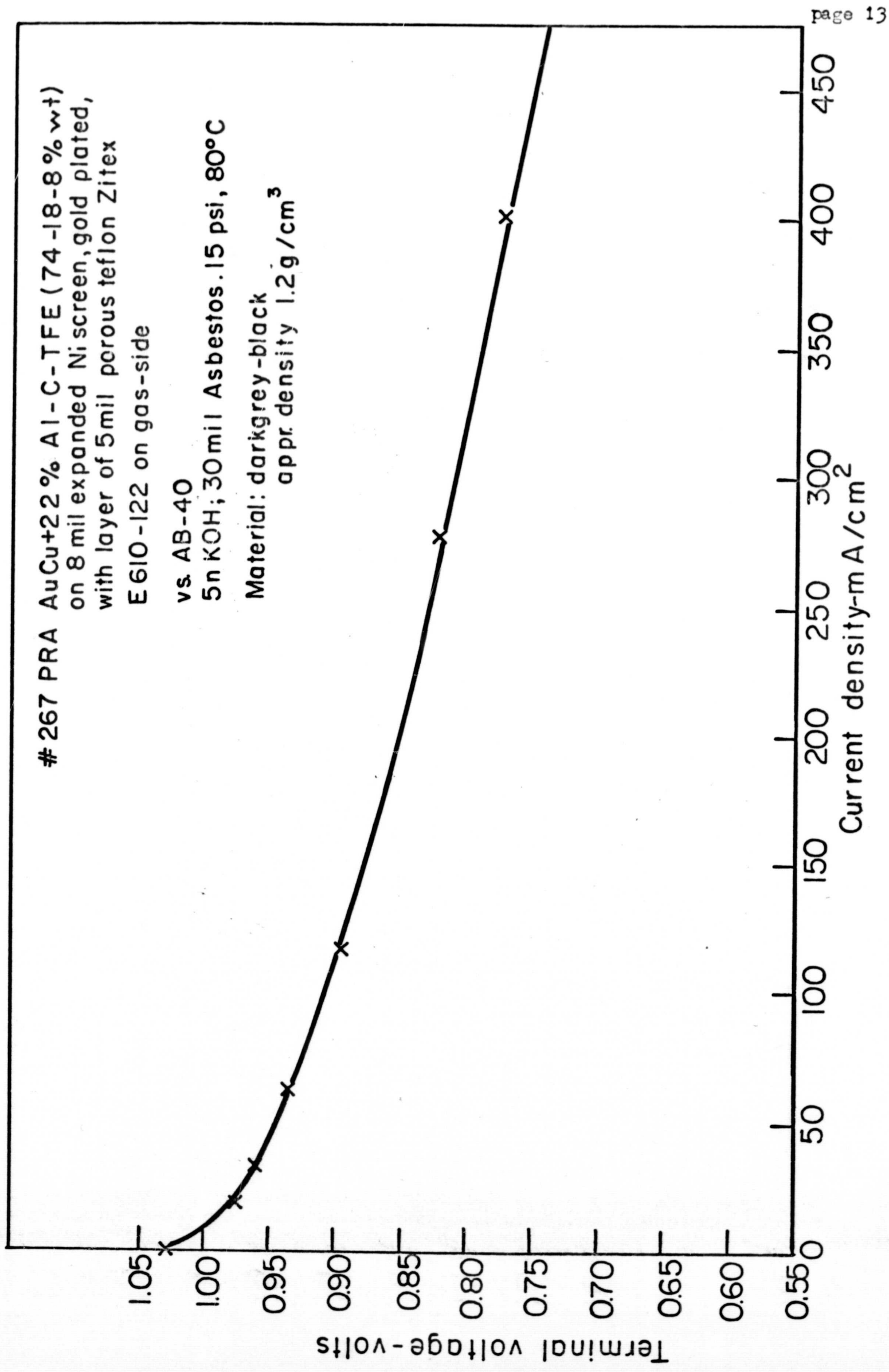


FIG. 2 -- Performance of early black with porous teflon

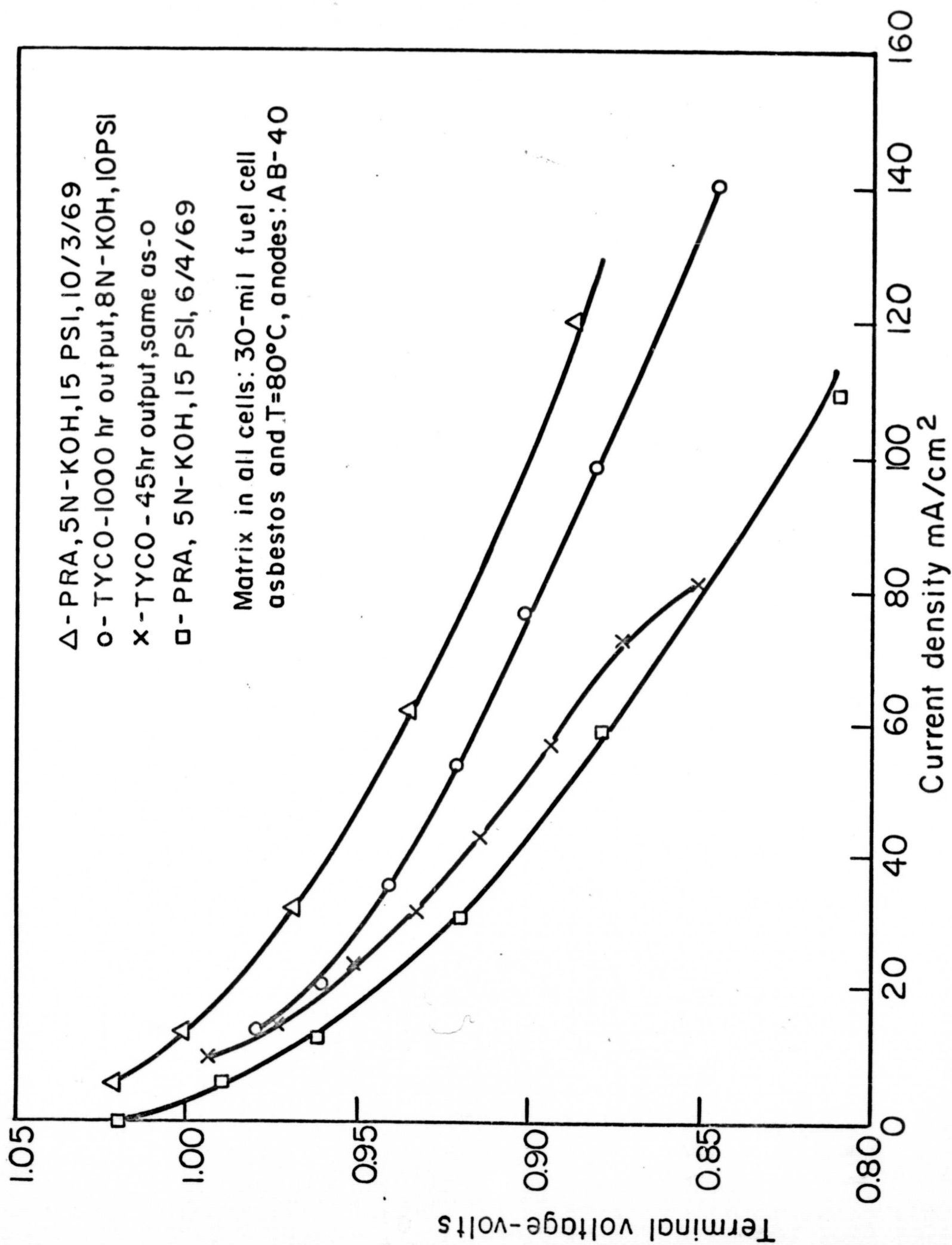


FIG. 3 -- H_2O_2 fuel cell electrode measurements: variations in AB-40 performance in oxygen reduction

Fig. 4 compares PRA and Tyco data* on their platinum-black cathodes. Again, note the striking change with time for the Tyco electrode. The similarity of behavior of a typical PRA electrode with that of a Tyco electrode suggests that independent investigators can produce reasonably similar electrodes with a given material in spite of different recipes. This figure also shows by dotted curves the extremes of the AB-40 performance shown in Fig. 3.

Fig. 5 shows performance of a typical PRA platinum black electrode as both cathode and anode. The two electrodes were AB-40 and a PRA aluminum-doped gold-copper crystalline cathode of brown color with a density of approximately 2.6 g/cm^3 . Similarity of the data suggests similarity of catalytic ability of the two platinum electrodes as anodes and our platinum and crystalline material as cathodes.

Fig. 6 shows data which compare the PRA incorporation of commercial powdered silver in one electrode, platinum black in another, and a PRA silver-aluminum alloy in a third. Although not as good as platinum, the silver alloy appears more satisfactory than pure silver.**

* "Development of Cathodic Electrocatalysts for Use in Low Temperature H₂/O₂ Fuel Cells with an Alkaline Electrolyte", Final Report covering the period 1 July 1965 to 30 June 1968, Contract NASW-1233 Q-12, Tyco Laboratories, pp. 172-186.

**This is not to be taken as an evaluation of the capabilities of doped crystalline Ag. The catalyst was coarsely ground and publication of this information is probably premature.

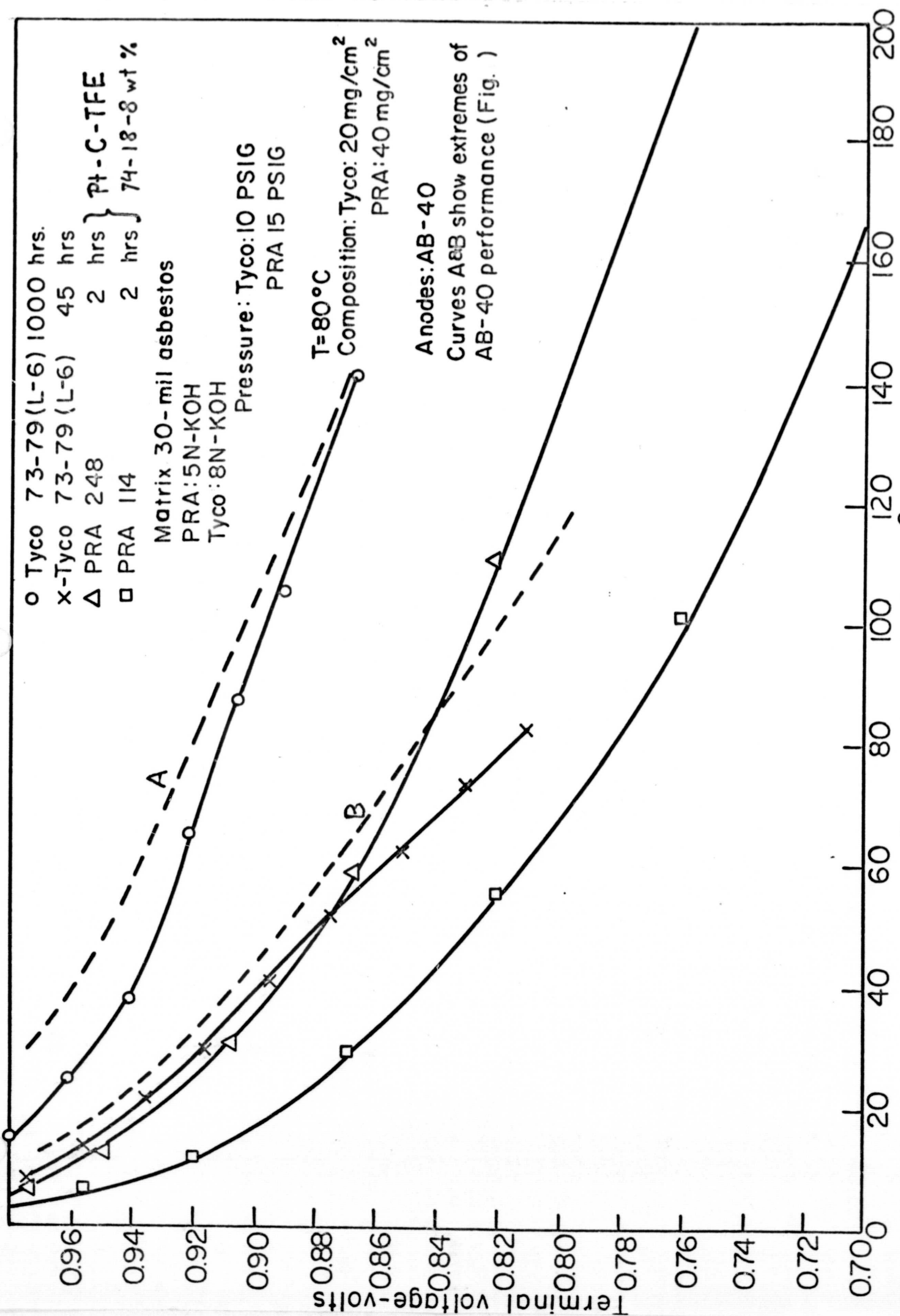


FIG. 4 -- H₂-O₂ fuel cell catalyst investigation; comparison of platinum electrodes for oxygen reduction

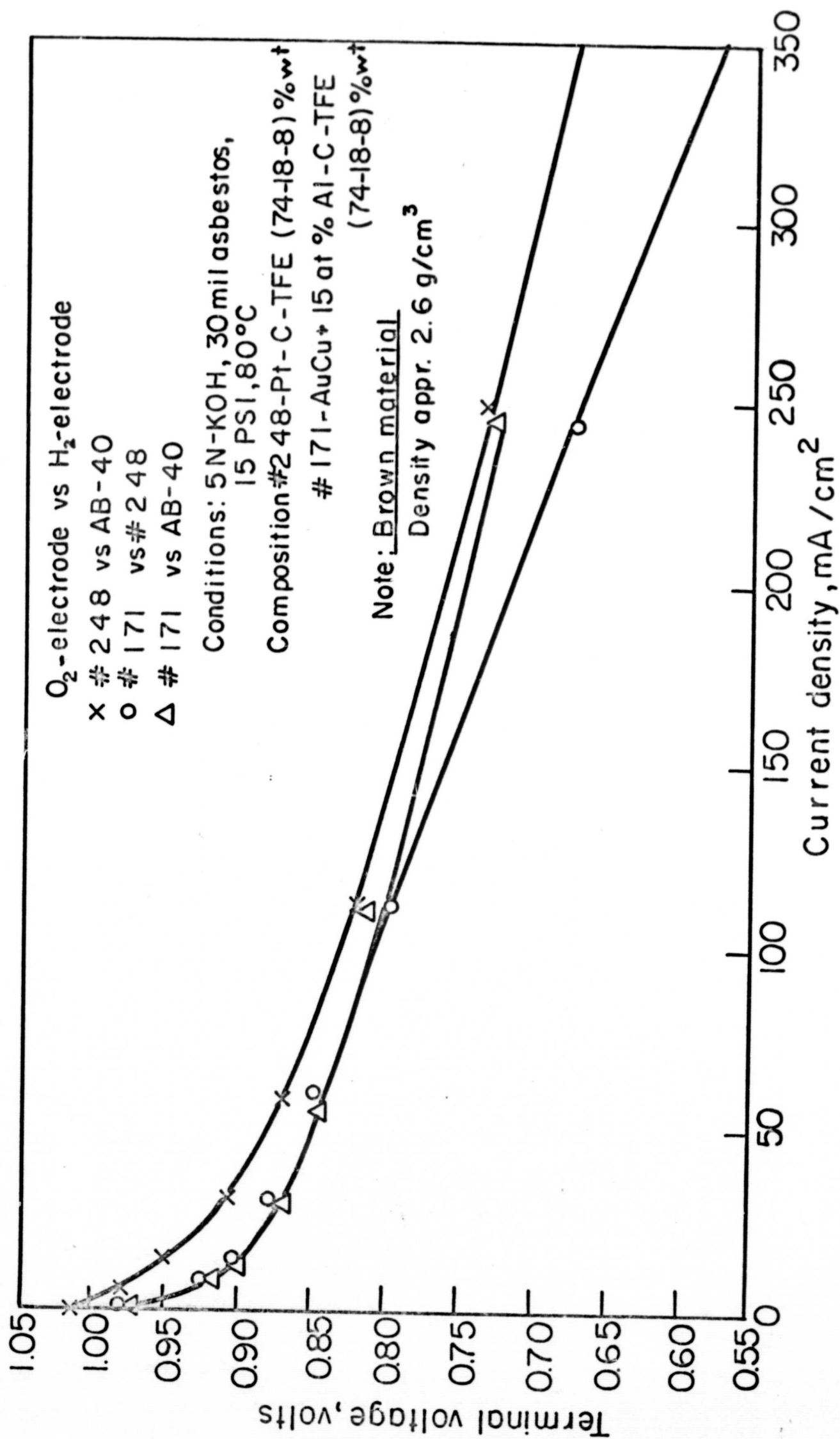


FIG. 5 -- Comparison of PRA Pt electrode performance
as cathode and anode

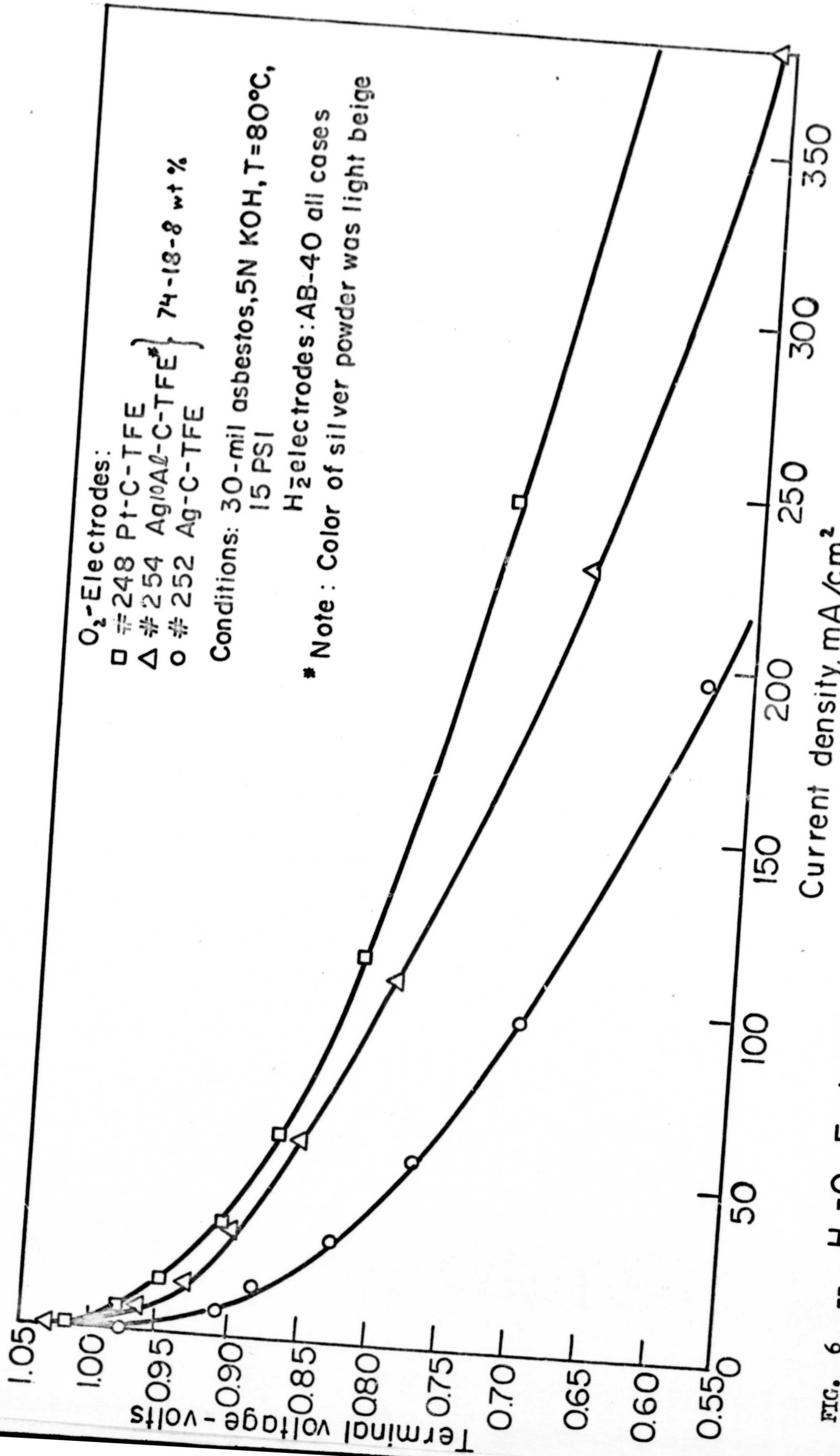


FIG. 6 -- H₂-O₂ Fuel cell catalyst investigation; PRA electrode comparison Pt vs. Ag

Fig. 7 shows typical data produced by doped gold-copper crystal powder. Data for curves 1 and 2 of Fig. 7 were taken during the period, and they duplicate previously reported results for these electrodes.

B.3.c PRA Electrodes as Anodes

The following information is about work not specifically required in the work statement. It is however reported as the selectivity of PRA catalyst is important. We anticipated that our catalyst as electron acceptors ought not to be active and expected therefore to find poor performance in electrodes which are used as anodes in H_2-O_2 fuel cells. Such selectivity is important in an oxygen electrode, as it greatly diminishes the possibility of explosions.

We have tested a number of our catalysts as anodes with AB-40 cathodes. Two representative cases are shown in Fig. 8. Noteworthy are both the low open cell voltages and poor current capabilities (effectively high electrode resistances) of these electrodes as anodes. We assume that the AB-40 electrodes were satisfactory cathodes, as the data in Fig. 3 show.

The difference in performance of these two electrodes as anodes does not appear to be significant.

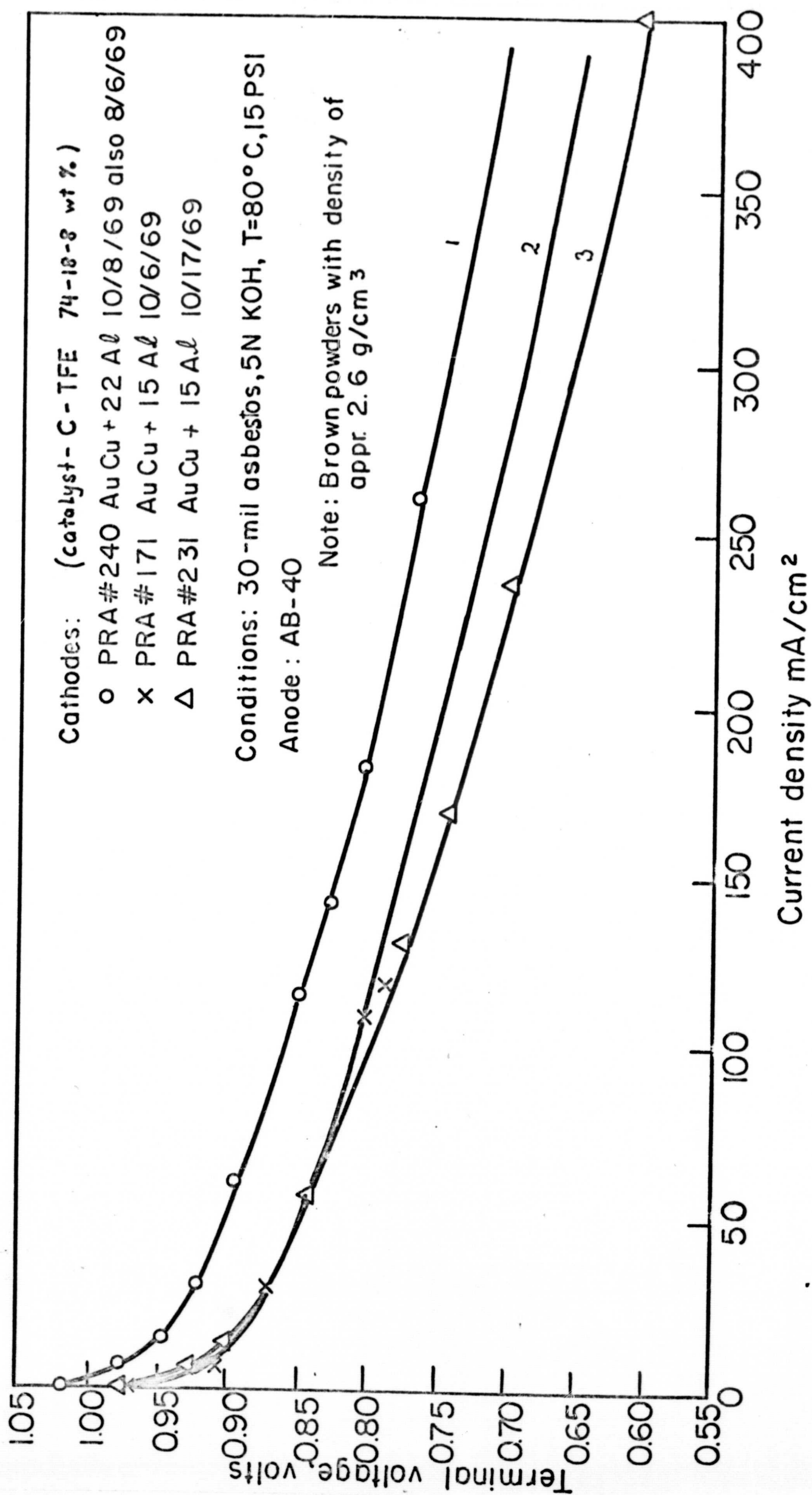


FIG. 7 -- Performance status of PRA catalysts for oxygen reduction

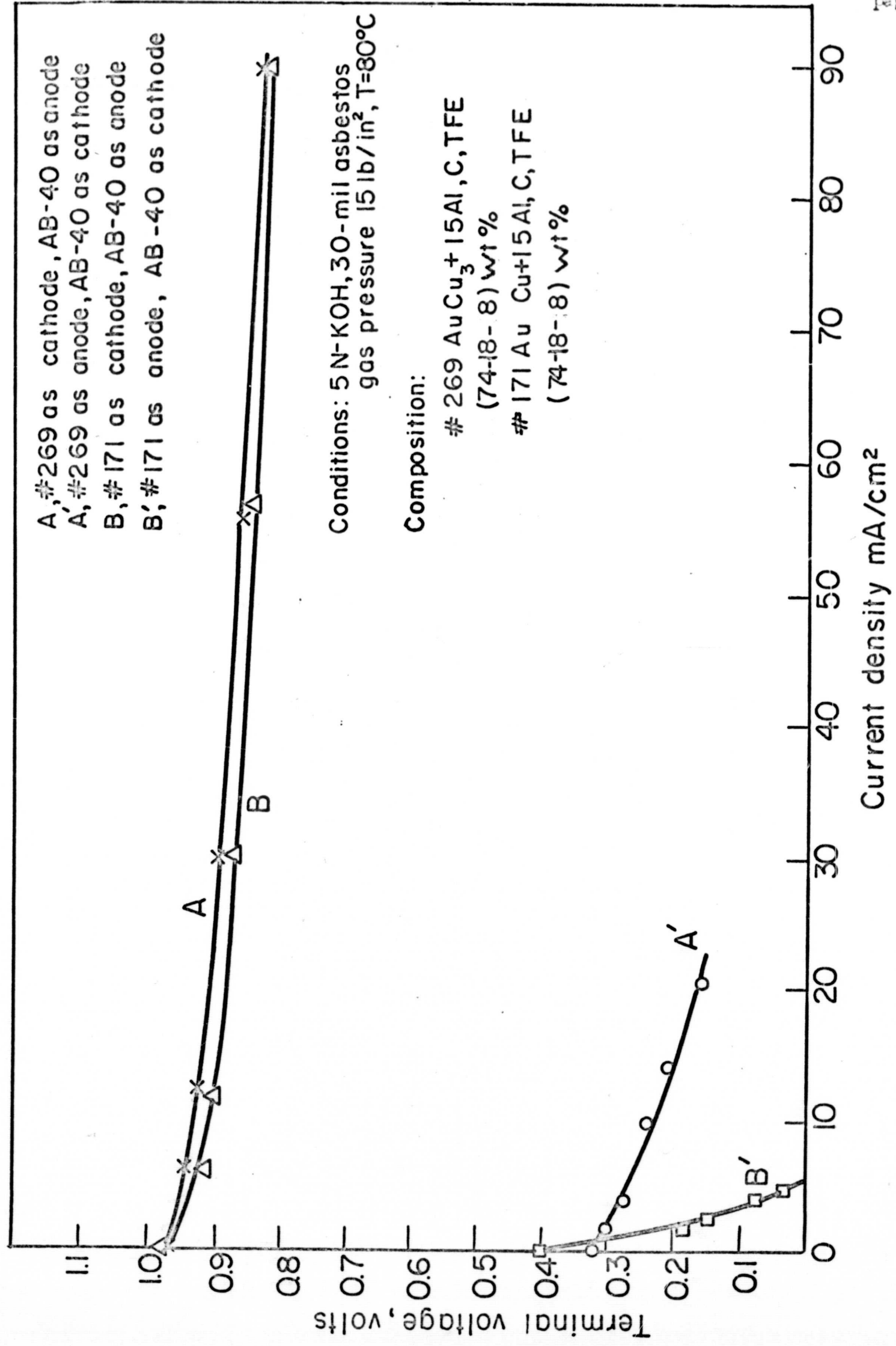


FIG. 8 -- Comparison of PRA catalysts as anodes and cathodes in H₂-O₂ fuel cells

B.4 Catalytic Performance as a Function of Particle Size

B.4.a Size Variation

We assume that the ground catalyst powders contain two particle groups which are different in size and in numbers. Two groups have been assumed.

The first group, the result of the first grinding, consists mainly of relatively large (5-10 μ) crystals, easily observed in the optical microscope.

The second group, the result of the second grinding in the attritor for about 72 hrs., can be seen only as diffraction discs in the optical microscope and must be assumed to be of the order of 0.5 μ or less.

We assume two populations, as follows:

<u>Population 2</u>		<u>Population 1</u>
size	1	d mean particle diameter*
population**	p	1
catalytic activity	p	d ²
weight	p	d ³

* expressed as the ratio of the larger particles to the smaller, the size of the smaller particles being considered unity.

** expressed as the ratio of the number of the more numerous to the less numerous, as above.

$$\text{Total catalytic activity} = p + d^2$$

$$\text{Total weight} = p + d^3$$

where catalytic activity is supposed to depend on the area of the particles, and the weight depends on volume. Then

$$\frac{\text{catalytic activity}}{\text{weight}} = \frac{p + d^2}{p + d^3}.$$

We are concerned with the variation of this ratio for constant weight.

If, for instance, $p = 10^2$, $d = 10$

$$1) \quad \frac{10^2 + 10^2}{10^2 + 10^3} = \frac{2 \times 10^2}{1.1 \times 10^3} \approx .18$$

If $p = 10^3$, $d = 10$

$$2) \quad \frac{10^3 + 10^2}{10^3 + 10^3} = \frac{1.1 \times 10^3}{2 \times 10^3} = .55$$

If $p = 10^4$, $d = 5$

$$3) \quad \frac{10^4 + 25}{10^4 + 125} = \frac{10^4}{10^4} \approx 1.$$

The ratio tends to unity for homogeneous samples. It is easy to show that these ratios are the catalytic activity per unit weight. One concludes that if, for instance, the mean population of the sample consists of particles of 0.5 μ diameter, a single 5 μ particle every 100 will reduce the catalytic activity to less than 1/5 that for a population of entirely small particles. One 5 μ particle in every thousand still reduces the potential catalytic activity of the sample to about $\frac{1}{2}$.

Thus the necessity for sufficiently efficient and prolonged grinding, followed by some system of separation of the fines, is evident. This difficulty does not exist for the blacks which are essentially homogeneous powders.

B.4.b Separation of Fines

A number of settling techniques are available for segregating solid particles of very small size. These techniques depend on the fact that heavy particles settle out first from a liquid when stirring stops. If all particles are of the same density, periodic drainage of precipitate results in sorting particles according to size.

Quantitatively, Stokes law is usually used

$$v = \frac{mg}{3\pi\eta r}$$

where

d is diameter of particle

m is mass of particle

g is acceleration due to gravity

η is coefficient of viscosity of the medium.

The particle mass is expressed in terms of its volume and the material density.

For our purposes, a liquid settling technique is undesirable because of the long settling times for the fines. There is further the disadvantage of drying the powder again after collection.

Therefore, a floatation technique also based on Stokes' law has been adopted. This procedure is a reversal of settling and uses floatation in a gaseous stream, where the finest particles are entrained first as stream velocity is increased. Assuming for a given flow velocity that spherical particles can be lifted and moved at uniform velocity with the stream, one may apply Stokes' theory directly. The coefficient of viscosity is that of the gas used, and the velocity is the particle's linear velocity, which is the ratio of volume flow (cc/min) to linear cross section of the enclosure (cm^2).

For our particular design, the Fischer-Porter Solv-Seal Teflon seal joints for glassware are being fabricated into a flow and collection assembly arranged in two vertical columns. Flows from 3 to 60 cc/min air will allow particles in various size ranges to be segregated, representing (for spherical particles) approximately 0.5 to 2.2 microns for the dimensions and apparatus chosen. As an example, the 3 cc/min flow will allow all particles less than 0.5 micron diameter to be collected, whereas quadrupling the flow velocity to 12 cc/min will allow particles of diameter up to 1 micron to be collected. (Note the square relationship between flow and diameter.)

The errors involved in applying this simple theory are primarily

- (1) that the fines cannot be assumed to be spherical, and
- (2) that agglomerates may form before, during, and after collection and in electrode fabrication.

There are the less important objections of variation in density, laminar flow, and perhaps incomplete collections of any given particle size.

On the basis of these objections we cannot claim accuracy of particle size in our electrodes except in the sense of equivalent particle size; that is, we can collect particles having aerodynamic behavior similar to 0.5-micron or 1-micron diameter spheres.

The components have been delivered, and tests should begin during the next month.

B.5 Life Test Fuel Cells

The life test cell that we are currently assembling is similar to that developed at Tyco Laboratories. Because of the nickel shortage, we were forced to make certain changes in design and some substitutions. We were able to make the cathode and anode plates of nickel, but other parts are of substitute materials.

A flange has been inserted as an intermediate piece between the anode plate and electrolyte pot assembly, made of # 304 stainless steel. The pot assembly is made of a stainless steel beaker (184 mm height - 155 mm diameter - capacity 3,300 ml, E. H. Sargent & Company cat. no. S-4860-7).

To attach the pot assembly to the flange required that a groove be cut in the pot assembly side of the flange. This groove was cut to accommodate two 1/8" X 1/8" square sided O-rings no. TS33-2607 obtained from Goshen Rubber Company, made of compound 1225, a 70 durometer

Buna N material. These O-rings were placed side by side to obtain a flat sealing surface. To hold the pot assembly to the O-rings a piece of 3/8" Al was rolled to fit on the under side of the lip of the beaker. Then a retaining disk of bakelite was cut with center holes .003" larger than the diameter of the pot assembly. This retaining disk has eight equally spaced holes to pass 10-24 screws. The flange plate is drilled and tapped to accommodate these screws. The electrolyte reservoir is thus clamped to the fuel cell and not welded. This ability to remove the pot assembly from the rest of the cell disposes of the need for a filler tube which is normally attached to the plate to which it is welded.

C. Discussion

By the end of this reporting period we should have achieved uniform particle size, i.e., below 0.5μ , by the use of new grinding techniques and air-flow particle separation. This is not necessarily either the smallest or the best particle size for optimal catalytic activity. Perhaps much work will remain to be done in this direction. But, for the time being this represents all we can do, and we shall stand on the basis of the results obtained from these powders.

As the consequences of inadequate grinding were not fully understood, it is difficult to evaluate the variations of electrode composition that have been tested in the past. These are being collated and should provide a valuable background to electrode fabrications using blacks exclusively.

Considerable work ~~also~~ remains to be done before our electrodes can be considered optimal. The techniques are possibly good enough to display the properties of the catalysts to advantage. During the next month a decision must be reached on this point, too, so that we can proceed with life testing. Life testing has been delayed for other reasons but should begin during December.

C.1 Improved Catalyst Chemical Resistance

We have discovered that occasionally some of our electrodes appear to have been attacked in the process of leaching out the calcium carbonate added for porosity purposes. We discovered on changing the acid medium that part of the difficulty lies in some leaching of copper. This copper apparently was not present in the alloy form which is impervious to chemical attack. Stoichiometry is carefully observed in making the catalysts, but our mixing operations may not be as thorough as formerly believed and some excess of copper may be present in some portions of the melt. The shot tower technique is in effect a detailed sampling of a melt by very small volumes. In crystallizing, excess copper (where present) would be forced out of the lattice and be found in interstitial positions and on the surface of the balls.

Although the possibility exists that Raney-type structures may result in our electrodes due to this leaching, experimental results indicate that excellence of electrode functioning does not depend on such actions:

- (1) because for many active samples such leaching, if it occurs at all, is beyond detection.
- (2) even where leaching takes place, no significant changes in performance are observed with time during the first few hours operation.

If anything, catalysts for which leaching has been observed are inferior originally to those free of this defect; their performance can generally be improved to the level of the latter by changing the KOH in the cell or by washing in acid media.

However, non-crystalline material should be removed from the fines, probably chemically. Some study of the problem is warranted in the sense of quantitative change in performance.

C.2 Improved Electrode Resistance

Improvement in conductivity of our materials will result from greater attention to annealing. Early work with thin films, which established the advisability of using ordered alloys as catalysts, was performed with carefully annealed specimens. Our shot tower method of preparing catalysts has tended to disregard this requirement, and our early success with ground samples caused us to overlook this extra refinement. However, with annealing the room temperature conductivity of ordered alloys increases by about a factor of 5* over that of the completely disordered state. Hence, we expect in the future to submit our fines to a suitable annealing cycle in an argon atmosphere.

In conclusion we expect that by use of the particle size separator, by removal of non-crystalline materials, and by annealing, improved and more consistent catalysts will be obtained.

* "The Modern Theory of Solids", by Frederick Seitz, First Edition, McGraw-Hill Book Co. Inc., 1940, pp. 40-41.

D. Plans for Next Quarter

We expect to prepare 200 grams of alloys divided in different batches of 25 grams each.

AuCu with 18 at-% In, Al -- one of each

AuCu with 22 at-% In, Al -- one of each

Ag with 18 and 22 at-% Al, In -- respectively

AgCu with 18 and 22 at-% In, Al -- respectively

These catalysts together with 7 at hand will be ground to fines less than 1μ , or when needed larger batches of alloys will be prepared, ground and separated using gas flotation until the required amounts of fines are obtained.

Three cathodes of 50 cm^2 area will be made of eight catalysts for a total of 24 of varying proportions of teflon and additives.

The catalyst will be mounted on expanded nickel meshes. The needed proportion of teflon is higher for very fine powder than for coarser ones and for the very open expanded nickel meshes than for woven meshes. The desired ratios of teflon and additives to catalyst have not yet been established.

Cathodes will be screened for life testing by initial performance, no less than 100 ma at 0.85 v after 48 hours, and eliminated if performance fails to improve during life test. Acceptable cathodes will be run at 400 ma/cm^2 and tested twice daily for performance at 100 ma/cm^2 . The conditions of test will be: electrolyte - 5 normal KOH, pressure - 15 psig, and temperature - 80°C . Each test will be run for up to two months. At the end of each test the electrolyte will be tested for traces of the cathodic alloy components.