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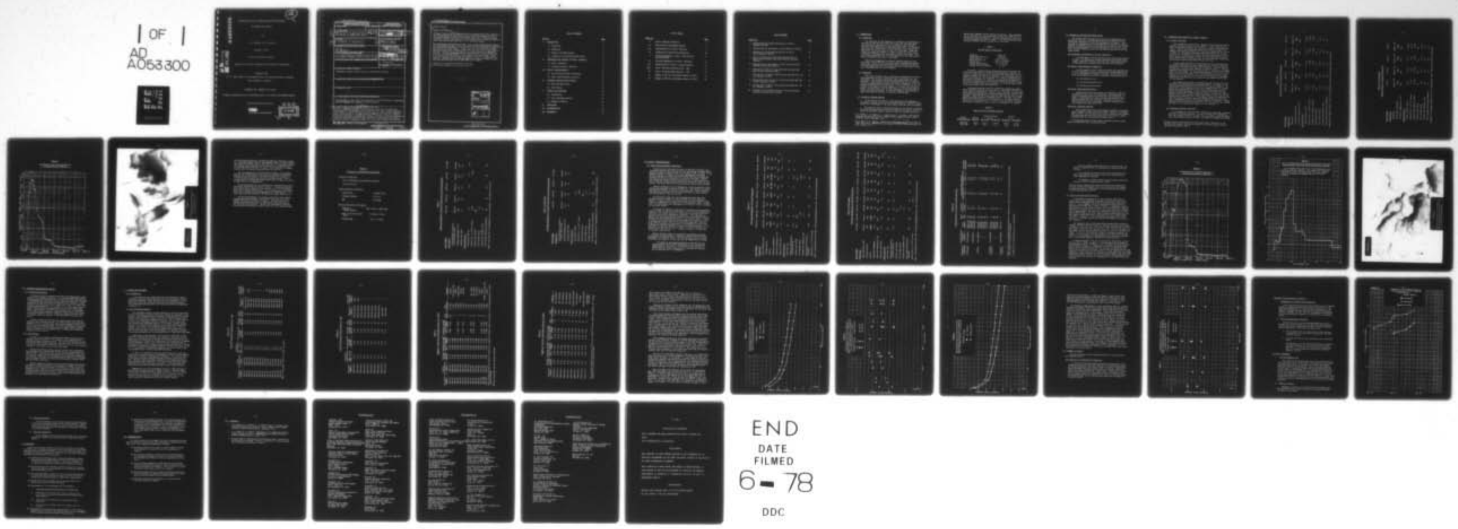

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OPTIMIZATION OF PT-DOPED KOCITE^{R} ELECTRODES IN H_3PO_4 FUEL CELLS

by

L. B. Welsh, R. W. Leyerle

January, 1978

Interim Technical Report

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The use of ~~UOP Inc.~~ Kocite^{R} electrocatalysts as low-cost air and/or fuel electrocatalysts in phosphoric acid electrolyte fuel cells is being optimized with respect to some of the electrocatalyst and electrode structure parameters. Kocite electrocatalysts are made from Kocite^{R} materials, which are composite structures consisting of pyropolymers chemically bonded to refractory substrates. Fuel cell electrodes are fabricated from these electrocatalysts and normally tested as anodes or cathodes in model fuel cells with standard platinum-black

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counter electrodes.

Different types of Kocite electrocatalysts were produced by first leaching of the alumina substrate from a Kocite material and then impregnating the resulting pyropolymer structure with platinum using one of several different techniques. Kocite electrocatalysts fabricated by these techniques normally have a 650 m^2/g surface area for a 15 wt-% platinum loading.

Teflon-bonded gas diffusion electrodes, with a porous graphite-paper backing were fabricated from these Kocite electrocatalysts by both machine-calendering and sheet molding techniques. Typical Kocite electrode platinum loadings were between 0.5 and 0.6 mg/cm^2. Model fuel cell test results were normally obtained at 180°C with air cathodes and H_2 anodes operated at atmospheric pressure. The best performance levels obtained using a Kocite cathode were 0.62 volts at 200 ma/cm^2 with a 65 mV oxygen gain at that current. With a cell having both a Kocite anode and cathode, 0.60 volts at 200 ma/cm^2 was achieved. Limited long term testing showed that Kocite electrodes can perform with good stability for at least 4500 hrs. at 180°C.

*"Kocite" is a registered trademark of UOP Inc.

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I. INTRODUCTION

1.1 Objectives

The objective of this program is to optimize the performance of low-cost air and fuel electrodes fabricated from platinum-impregnated UOP Kocite materials for use in phosphoric acid electrolyte fuel cells operating at temperatures near 180°C. To accomplish this objective, various Kocite electrocatalysts are produced and fabricated into state-of-the-art fuel cell electrodes. These electrodes are tested as cathodes or anodes in model fuel cells using conventional platinum black electrodes as counter electrodes. The cell testing determines both the performance level and long term endurance attainable with Kocite electrodes.

This report summarizes the results obtained during the last nine months. Both the Kocite electrocatalyst formulations prepared during this period and the performance levels attained with Kocite electrodes made from these electrocatalysts will be discussed, along with recommendations for future research.

1.2 Background

The current research effort continues the optimization of Kocite electrocatalysts for use in phosphoric acid electrolyte fuel cell electrodes. The program organization established earlier is being continued. This involves a joint program with UOP Inc. as prime contractor and Energy Research Corporation (ERC, a subsidiary of St. Joe Minerals Corporation) as subcontractor. A broad division of effort is made on the basis of the UOP capability to produce Pt-impregnated Kocite electrocatalysts and the ERC capability to fabricate and optimize electrode structures. Testing and diagnostic evaluations of these electrodes are carried out jointly by UOP and ERC.

1.3 Review of Previous Results

In this section the status of this program at the beginning of the present contract period will be briefly reviewed to provide a background for a discussion of the effort made during the last nine months.

The nature of Kocite materials and many of the process conditions for preparing these materials have been described in earlier reports.[1,2] During the previous contract periods a number of different Kocite ma-

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[1] L. B. Welsh, R. W. Leyerle, G. L. Hervert and K. J. Youtsey, "Carbonaceous Catalysts for H_3PO_4 Fuel Cells", MERDC Contract DAAK02-75-C-0011, Final Technical Report, September, 1975.

[2] L. B. Welsh, R. W. Leyerle, "Optimization of Pt-Doped Kocite^{R} Electrodes in H_3PO_4 Fuel Cells", MERADCOM Contract DAAG53-76-C-0014, Interim Technical Report, January, 1977.

terials were examined as electrocatalyst supports. This evaluation led to the choice of a Kocite material using Alcoa's Hydral 705 as the starting substrate material. Revelant properties of this type of Kocite material are given in Table I. All Kocite materials used during the present contract period are of this type.

Table I

Kocite^R Material Properties

Substrate	Hydral 705
Pyropolymer Precursor	Benzene
Carbon Content	20 to 26 wt-%
Resistivity at 25°C	0.08 to 0.20 Ω-cm
Surface Area	80 to 120 m^2/g
Average Pore Diameter	5.5 to 6.0 nm
Average Particle Diameter	0.5 μm

All Kocite electrocatalysts prepared during the previous contract period were made by the platinum impregnation of Kocite materials.[2] Toward the end of that period it was ascertained that the alumina substrate was leached by phosphoric acid from small Kocite particles during fuel cell operation. As a result, experiments were initiated to determine the properties of leached Kocite materials. The initial preparations of these leached materials indicated a high surface area ($\gtrsim$700 m^2/g) was attainable[2], but no Kocite electrocatalysts were prepared from these materials.

A substantial Kocite electrode optimization program was undertaken during the previous contract period. Variations in Teflon content, catalyst layer density, catalyst layer thickness, and electrode prewetting (with H_3PO_4) were studied and numerous cells tested at UOP and ERC.[2] Generally ERC's Kynol matrices were used but one cell was assembled using a SiC matrix prepared by ERC.[2] The best cell performance results obtained up to the inception of the present contract period are listed in Table II.[2] For both cells the Kocite electrode was used as a cathode at 180°C and had a Pt-loading of $\sim$0.5 mg/cm^2.

Table II

Best 1976 Cell Performance Results

Kocite Electrocatalyst	Matrix Material	Terminal Voltage on air		O_2 Gain at	
		100 ma/cm^2	200 ma/cm^2	100 ma/cm^2	200 ma/cm^2
3380-156G	Kynol	0.65 V	0.565 V	60 mV	70 mV
3380-156G	SiC	0.66 V	0.57 V	70 mV	100 mV

1.4 Program for the Past Nine Month Period

The primary effort during this period was the preparation and evaluation of Kocite electrocatalysts made from leached Kocite materials and catalyzed by various Pt-impregnation techniques. This required an optimization of the leaching process by which the alumina substrate is leached from the Kocite material by H_3PO_4 as well as substantial characterization of the leached materials by various techniques. The main efforts of the last nine months have been in the following areas:

(A) Preparation and Leaching of Kocite Materials

A large number of Kocite materials were prepared and leached in H_3PO_4. By varying the leaching conditions and examining the product, desirable leaching conditions were established by which a high-surface-area leached Kocite material can be obtained.

(B) Kocite Electrocatalyst Preparation

A large number of Kocite electrocatalysts were prepared both by first leaching the Kocite material and then impregnating with Pt or Pt-impregnation followed by leaching. Kocite electrocatalysts were prepared by different techniques involving the use of

(1) Chloroplatinic Acid,

(2) Platinum Diaminedinitrite,

(3) Platinum Acetylacetonate.

(C) Kocite Electrode Fabrication

Several Kocite electrocatalysts were delivered to ERC for incorporation into fuel cell electrodes. Several machine calendered electrode batches were fabricated for testing and a number of experimental electrode batches fabricated by a sheet molding process were also prepared. During this last nine month period less effort was placed on Kocite electrode structure optimization than during 1976.

(D) Kocite Electrode Testing

Kocite electrodes were tested almost exclusively as cathodes using 2 mg/cm^2 Pt black counter electrodes. The cell performance goal of 0.600 V at 200 ma/cm^2 with a Kocite cathode of ∿0.5 mg/cm^2 Pt-loading was met with cells operating on air and H_2 at 180°C. Endurance data were accumulated on several cells.

A detailed discussion of the results obtained in each of these areas is presented in the following sections.

II. PREPARATION AND LEACHING OF KOCITE MATERIALS

2.1 Kocite Materials

The preparation of the Kocite materials used during the present contract period is reported in this section. In all cases Alcoa's Hydral 705 alumina substrate was used with a benzene pyropolymer precursor. Some of the preparation conditions as well as some of the chemical and physical properties of these Kocite materials are listed in Table III. Equipment replacement and recalibration led to some variation in the properties of Kocite materials made during the early part of this contract period, however, with the exception of Kocite batch 3289-195, Kocite materials used for electrocatalyst preparation had starting surface areas between 82 and 124 m^2/g.

A pore volume distribution was obtained on two Kocite materials by nitrogen adsorption for later comparison with the pore volume distribution of the resulting Kocite electrocatalyst. The pore volume distribution of Kocite batch 3289-120 is shown in Figure 1. The most important characteristic of this distribution is the relatively narrow range of pore volumes. The distribution maximum occurs at 4.0 nm diameter pores and two shoulders in the distribution are found at pore diameters of 8.0 and 12.5 nm. For this Kocite material the dominant surface area contributions come from pores with diameters less than ∿5.0 nm diameter.

An attempt was also made to characterize the structure of Kocite particles by Scanning Electron Microscopy (SEM).[3] An example of the results obtained with Kocite batch 3576-60 is shown in Figure 2. In this figure, Kocite powders are shown at a magnification of 100,000X. Based on this micrograph and others, Kocite powders made with the Hydral 705 substrate appear to have a platelet structure with many particles having their largest dimension smaller than 200 nm. Micrographs of this type not only indicate that further study of Kocite materials and electrocatalysts at these magnifications are warranted, but that the platelet structure and proper sample preparation may allow useful Transmission Electron Micrographs (TEM) to be obtained. Higher magnification studies with TEM may allow the pore structure not visible in Figure 2, but indicated by Figure 1, to be resolved.

2.2 Leaching of Kocite Materials

To determine the most desirable conditions for leaching the alumina substrate from Kocite material, a variety of small batch experiments were run in which the ratio of H_3PO_4 to Kocite powder, leaching temperature, and leaching time were varied. The range of parameter variations

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[3] Pictures taken at Scanning Electron Microscope Users' Laboratory at the University of Chicago. The lab is operated on a resource grant from the National Institutes of Health.

Table III

Characteristics of Kocite^R Materials

Batch Number	3289-120	3289-140	3289-195	3576-10	3576-20
Preparation					
Substrate	Hydral 705	Hydral 705	Hydral 705	Hydral 705	Hydral 705
Pyropolymer Precursor	C_6H_6	C_6H_6	C_6H_6	C_6H_6	C_6H_6
Pyrolysis Temperature (°C)*	840	870	830	810	840
Characteristics					
Carbon Content (Wt-%)	21.52	26.22	27.35	23.8	26.62
Resistivity, ρ, @ 25°C (Ω-cm)	0.17	0.22	0.086	0.183	0.080
Apparent Bulk Density (g/ml)	0.30	0.30	0.267	0.288	0.265
Surface Area (m^2/g)	107	122	38	124	77
Pore Volume (ml/g)	0.15	0.17	0.06	0.15	0.11
Average Pore Diameter (nm)	5.6	5.6	6.3	4.8	5.7
Pore Volume Distribution Measured	Yes	Yes	No	No	No

*Temperature changes caused in part by thermocouple repair and repositioning at mid-bed.

Table III (Continued)

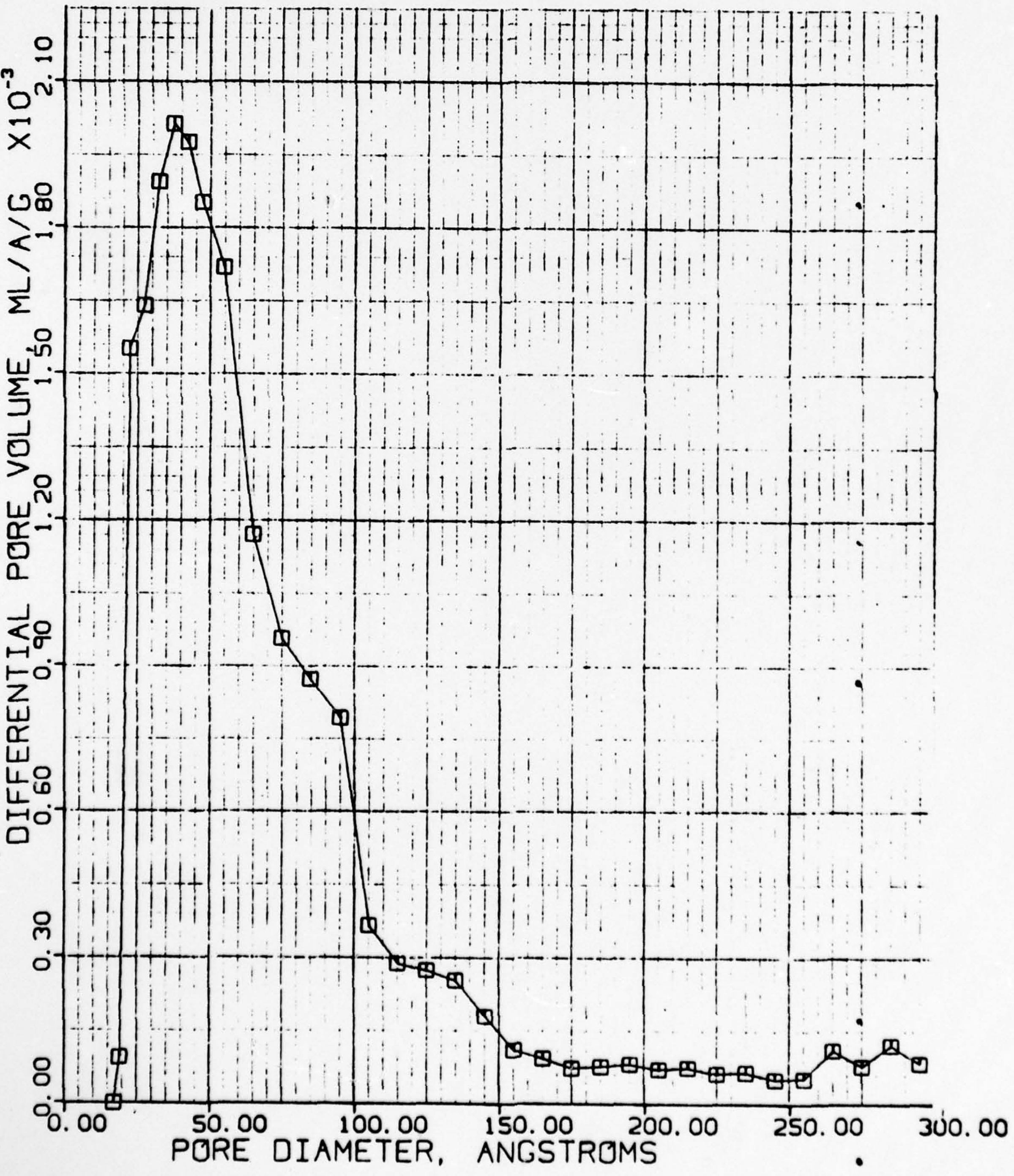
Characteristics of Kocite^{R} Materials

Batch Number	3576-40	3576-60	3576-65	3576-70
Preparation				
Substrate	Hydral 705	Hydral 705	Hydral 705	Hydral 705
Pyropolymer Precursor	C_6H_6	C_6H_6	C_6H_6	C_6H_6
Pyrolysis Temperature (°C)*	840	865	870	865
Characteristics				
Carbon Content (Wt-%)	24.17	25.33	24.77	22.92
Resistivity, ρ, @ 25°C (Ω-cm)	0.12	0.14	0.14	0.13
Apparent Bulk Density (g/ml)	0.290	0.278	0.278	0.292
Surface Area (m^2/g)	94	123	82	88
Pore Volume (ml/g)	0.14	0.17	0.11	0.12
Average Pore Diameter (nm)	6.0	5.5	5.4	5.5
Pore Volume Distribution Measured	No	No	No	No

*Temperature changes caused in part by thermocouple repair and repositioning at mid-bed.

Figure 1

Differential Pore Volume Distribution of Kocite Material 3289-120



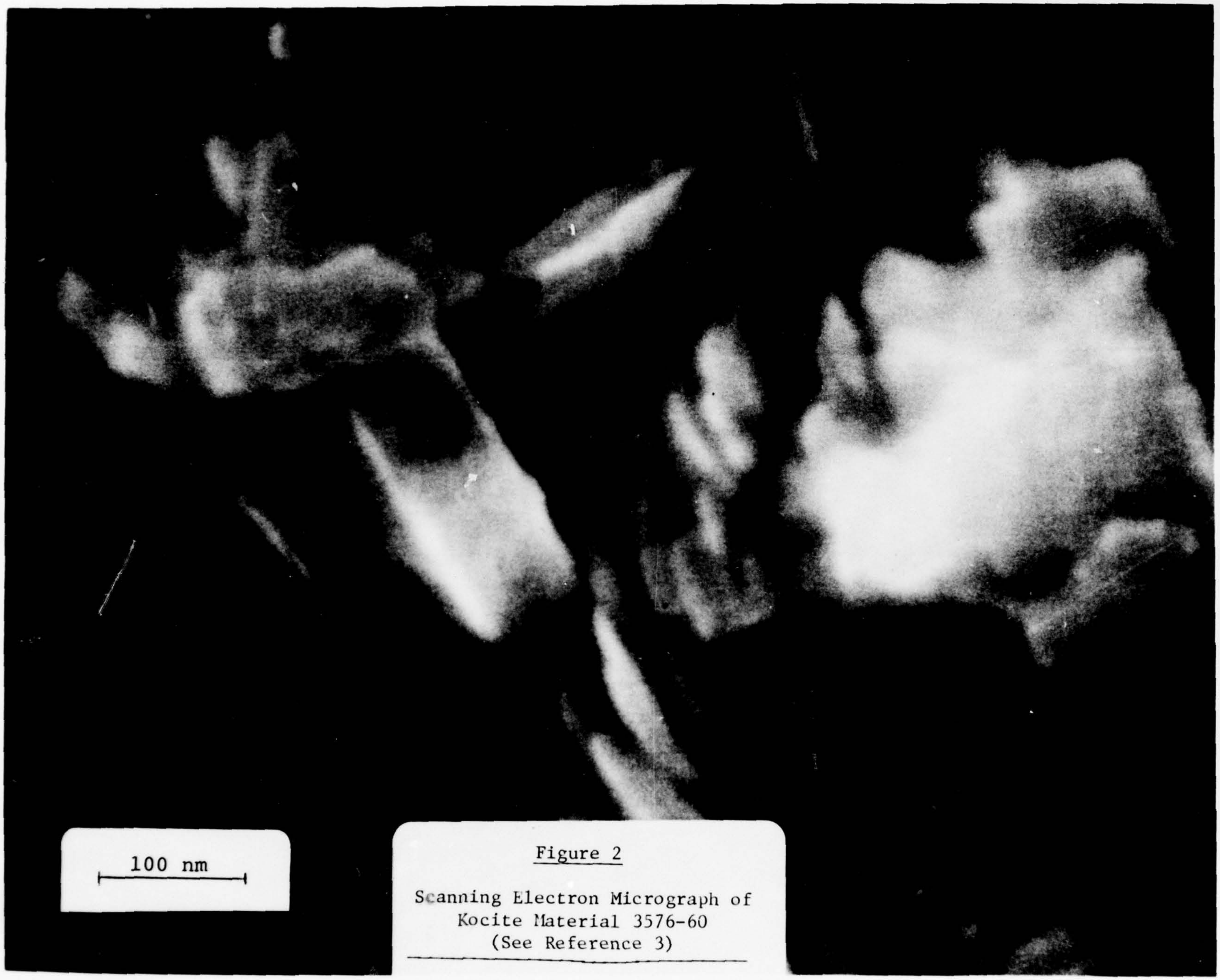


Figure 2

Scanning Electron Micrograph of
Kocite Material 3576-60
(See Reference 3)

and the leaching conditions considered optimal are indicated in Table IV. These experiments indicate that desired product of Kocite material starting with a surface area of $\sim 100\ m^2/g$ and a 25 wt-% carbon content is a leached Kocite material with a surface area of $\sim 800\ m^2/g$ and a 0-5 wt-% ash content relative to carbon. Such a material is readily obtained with the process conditions given in Table IV.

For the preparation of electrocatalyst supports, the equipment used for the leaching process was scaled up to handle 250 g Kocite batches (50-70 g of product). The Kocite powders are leached in a 5 ℓ heated flask and stirred during the leaching process. The resultant product is washed and dried several times to remove all traces of H_3PO_4 prior to Pt impregnation.

The characteristics of the leached Kocite materials used in electrocatalyst preparation are listed in Table V. In general, leached Kocite materials are denoted by the addition of an "L" following the Kocite batch identification number. In two cases Kocite electrocatalysts were prepared by leaching subsequent to Pt-impregnation. In these cases the electrocatalyst identification number ends in an "L".

For selected materials, ash content, resistivity and a surface area analysis were performed. Ash contents less than 5 wt-% were normally obtained as indicated in Table V. The resistivity of a leached Kocite material showed no significant change from that of the starting Kocite material. Surface area measurements indicated that leached Kocite material surface areas are in the expected range.

Table IV

Production of Leached Kocite Materials

Production Conditions

10 ml of 85% H_3PO_4 per gram Kocite material

140°C for 24 hrs.

Characterization of Material

Surface Area	750-1000 m^2/g
Inorganic Residue	0-5 wt-%
ABD	0.05 g/ml

Leaching Variations Investigated

Temperature (water content)	100°C (15%) to 180°C (0%)
H_3PO_4 to Kocite material Ratio	25 ml/g to 5 ml/g
Leaching Time	8 hr. to 4 days

Table V

Leaching Parameters of Kocite^{R} Materials and Electrocatalysts

Batch Number	3289-120L	3289-140L	3289-195L	3576-10L	3576-20AL
Preparation					
Kocite Material or Electrocatalyst Batch Used*	3289-120	3289-140	3289-195	3576-10	3576-20A
Leaching Temperature (°C)	140	140	141	142	143
Leaching Time (Hrs)	24	22	24	24	24
Characterization					
Ash Content (Wt-%)	4.9		0.0	2.0	0.0
Resistivity, ρ, @ 25°C (Ω-cm)	0.107			0.107	
Surface Area (m^2/g)	989				
Pore Volume (ml/g)	1.76				
Average Pore Diameter (nm)	7.1				
Pore Volume Distribution Measured	No	No	No	No	No

*Batch 3576-20AL was Pt impregnated prior to leaching. See Tables VI and VII.

Table V (Continued)

Leaching Parameters of Kocite^{R} Materials and Electrocatalysts

Batch Number	3576-44AL	3576-60L	3576-65L	3576-70L
Preparation				
Kocite Material or Electrocatalyst Batch Used*	3576-44A	3576-60	3576-65	3576-70
Leaching Temperature (°C)	141	142	145	143
Leaching Time (Hrs)	24	24	24	24
Characterization				
Ash Content (Wt-%)		4.8		
Resistivity, ρ, @ 25°C (Ω-cm)		0.166		
Surface Area (m^2/g)		881		
Pore Volume (ml/g)		1.57		
Average Pore Diameter (nm)		7.1		
Pore Volume Distribution Measured	No	No	No	No

*Batch 3576-44AL was Pt impregnated prior to leaching. See Tables VI and VII.

III. KOCITE ELECTROCATALYSTS

3.1 Kocite Electrocatalyst Preparation

During the last nine month contract period about 40 Kocite electrocatalyst batches were prepared from the leached and unleached Kocite materials listed in Tables IV and V, including a number of small (<10 g) batches prepared to evaluate the various impregnation techniques. Several larger Kocite electrocatalyst batches have been prepared and delivered to ERC for incorporation into Kocite electrodes. Some of the preparation details and physical properties of electrocatalysts sent to ERC are listed in Tables VI and VII, along with those of other batches from which significant characterization results were obtained.

Details relating to the Pt-impregnation of Kocite materials (both leached and unleached) are given in Table VI. Also listed are surface area analysis results on selected electrocatalysts. Pore volume distributions were obtained for two electrocatalysts and these results will be discussed in the next section.

As indicated in Table VI, most impregnations were made using chloroplatinic acid (CPA) with either ethanol or water solvents. However, a number of Kocite electrocatalysts were also prepared using either Pt diaminedinitrite (PDD) or Pt acetylacetonate (PAA). In general, electrocatalysts were impregnated at a level of ~ 15 wt-% relative to the carbon content.

To discuss the physical properties of the Kocite electrocatalysts listed in Table VI and the relative merits of the various impregnation techniques, the characteristics of these Kocite electrocatalysts are listed in Table VII by impregnation technique. Also indicated is whether the electrocatalyst was prepared by first leaching the Kocite material and then impregnating with Pt or the reverse order. In Table VI results are given as well for electrocatalyst Pt crystallite size as determined by X-ray diffraction line width broadening. For the larger (60 g) batches various steps of the impregnation process were performed in different equipment than for the smaller batches. It is believed that this may have led to larger Pt crystallites being obtained with larger batches due to inadequate mixing; a problem that is currently being solved.

Several different types of catalyzation experiments were made to determine the best method for producing a Kocite electrocatalyst with small Pt crystallite size. The most important results of these experiments are apparent in Table VII and are summarized as follows:

(A) Using the CPA/C_2H_5OH impregnation technique several electrocatalyst batches were prepared by either leaching then impregnating or vice versa. Pt crystallite size results indicate that more highly dispersed Pt is obtained by leaching Kocite material prior to impregnation.

Table VI

Platinum Impregnation of Kocite^R Materials

Batch Number	3289-127A	3289-144A	3289-144C	3289-197A	3576-12A	3576-12C	3576-12E	3576-12F
Preparation								
Kocite Material Used	3289-120L	3289-140L	3289-140L	3289-195L	3576-10L	3576-10L	3576-10L	3576-10L
Platinum Source*	CPA	CPA	PAA	CPA	PDD	PDD	CPA	CPA
Solvent	C_2H_5OH	C_2H_5OH	C_6H_6	C_2H_5OH	30% HNO_3	30% HNO_3	H_2O	H_2O
Reduction Atmosphere	H_2	H_2	N_2	H_2	N_2	N_2	H_2	H_2
Reduction Temperature (°C)	260	260	260	260	260	260	260	260
Reduction Time (Hrs)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Characteristics								
Batch Size (g)	60	60	5	60	2	5	10	10
Platinum Concentration, Pt/(Pt+C) (Wt-%)	17.3	15	15	15	15	14.8	15	5
Surface Area (m^2/g)	661	610				642		
Pore Volume (ml/g)		1.08				0.81		
Average Pore Diameter (nm)		7.1				5.0		
Pore Volume Distribution Measured	Yes	Yes	No	No	No	No	No	No
Resistivity, ρ, @ 25°C (Ω-cm)								

*CPA = chloroplatinic acid; PDD = platinum diaminedinitrite, PAA = platinum acetylacetonate.

Table VI (Continued)

Platinum Impregnation of Kocite^R Materials

Batch Number	3576-20AL	3576-44AL	3576-61A	3576-61B	3576-61C	3576-61D	3576-61E	3576-61F	3576-66B
Preparation									
Kocite Material Used	3576-20	3576-40	3576-60L	3576-60L	3576-60L	3576-60L	3576-60L	3576-60L	3576-60L
Platinum Source*	CPA	CPA	PDD	CPA	CPA	CPA	CPA	PDD	CPA
Solvent	C_2H_5OH	C_2H_5OH	H_2O	H_2O	H_2O	H_2O	H_2O	H_2O	H_2O
Reduction Atmosphere	H_2	H_2	H_2	H_2	H_2	H_2	H_2	N_2	H_2
Reduction Temperature (°C)	260	260	260	260	260	260	260	260	260
Reduction Time (Hrs)	2.0	2.0	2.0	2.0	2.0	1.5	2.5	2.0	2.0
Characteristics									
Batch Size (g)	60	60	10	10	10	10	10	10	30
Platinum Concentration, Pt/(Pt+C) (Wt-%)	14.9	15	11.4	16.3	15	15	15	15	15
Surface Area (m^2/g)			620						
Pore Volume (ml/g)			1.07						
Average Pore Diameter (nm)			6.9						
Pore Volume Distribution Measured	No	No	No	No	No	No	No	No	
Resistivity, ρ, @ 25°C (Ω-cm)				0.15					

*CPA = chloroplatinic acid; PDD = platinum diaminedinitrite, PAA = platinum acetylacetonate.

Table VII

Characteristics of Kocite^R Electrocatalysts

Impregnation Technique	Electrocatalyst Preparation Method	Batch Number	Batch Size (g)	Platinum Concentration (Wt-%)	Platinum X-Ray Analysis		Electrodes Fabricated
					Agglomeration[a] (%)	Crystallite Size[b] (nm)	
CPA/C_2H_5OH	Leach-Impreg	3289-127A	60	17.3	37	6.7	Yes
	"	3289-144A	60	15	43	11.7	No
	"	3289-197A	60	15	40	10.6	No
	Impreg-Leach	3576-20AL	60	14.9	57	9.2	Yes
	"	3576-44AL	60	15	54	7.4	No
CPA/H_2O	Leach-Impreg	3576-61B	10	16.3	25	5.2	Yes
	"	3576-61C	10	15	31	7.5	No
	"	3576-61D	10	15	12	5.5	No
	"	3576-61E	10	15	49	8.0	No
	"	3576-12E	10	15	24	8.5	No
	"	3576-12F	10	5	31	14.0	No
Pt Diamine-dinitrite	Leach-Impreg	3576-12A	2	15	22	1.3	No
	"	3576-12C	5	15	8	9.6	No
	"	3576-61A	10	15	9	4.7	Yes
	"	3576-61F	10	15	~0	-	No
	"	3576-66B	30	15	62	13.6	No
Pt Acetyl-acetonate	Leach-Impreg	3289-144C	5	15	69	8.4	No

[a] These results are standardized as discussed in Reference 1 except that Kocite electrocatalyst 3576-66A (not listed) is used as the standard.

b Mean size of crystallites over 2.5 nm diameter.

(B) Both C_2H_5OH and H_2O solvents were tried with CPA. In general, slightly better Pt dispersions are obtained with H_2O solvents.

(C) Pt crystallite sizes on Kocite electrocatalysts prepared using PDD are much smaller than those obtained by any other impregnation technique.

(D) Attempts to prepare Kocite electrocatalysts using PAA have met with only limited success to date.

With the results obtained from these electrocatalyst preparations, the data of Table VII suggest the electrocatalyst with the highest Pt dispersion should be obtained by first leaching Kocite material and then impregnating with PDD.

3.2 Kocite Electrocatalyst Structure

A preliminary determination of the structural properties of several Kocite electrocatalysts was made in addition to the structural property determinations of Kocite materials discussed in section 2.1. As an example, the pore volume distribution of Kocite electrocatalyst 3289-127A is shown in Figure 3. Comparison of this distribution with that shown in Figure 1 for Kocite material 3289-120 indicates the changes occurring in the pore sizes both upon leaching and Pt impregnation. The peak in the PVD shifts from 4.0 to 7.0 nm and the pore volume at all pore sizes increases substantially for the much higher surface area Kocite electrocatalyst (on a weight basis). In Figure 3, shoulders in the distribution are apparent at 3.0 nm and 12.5 nm. Similar results were obtained with Kocite electrocatalyst 3289-144A.

The changes in pore volume distribution between Kocite electrocatalyst 3289-127A and Kocite material 3289-120 are most clearly shown in Figure 4, where the ratio of the pore volume distributions is plotted. This figure shows that there has been more than a 15-fold increase in the number of pores having diameters between 7.5 and 10.0 nm, while the number of pores at other diameters shows a substantially smaller increase. It is believed that these changes in the PVD from Kocite material to electrocatalyst are determined by the structural configuration of the alumina substrate during pyropolymer deposition.

An SEM micrograph[3] of Kocite electrocatalyst 3576-61B on an electrode surface is shown in Figure 5. This micrograph was obtained at the same magnification, 100,000X, as the micrograph of Kocite material 3576-60 shown in Figure 2. Comparison of these two micrographs suggests that the gross catalyst particle structure is similar to the initial Kocite particle structure. Further SEM and TEM studies will be required to fully ascertain the similarity of these materials, but these preliminary results coupled with the PVD results suggest that the carbonaceous pyropolymer structure is neither destroyed nor substantially altered by either the leaching, Pt impregnation or electrode fabrication processes.

Figure 3

Differential Pore Volume Distribution of Kocite Electrocatalyst 3289-127A

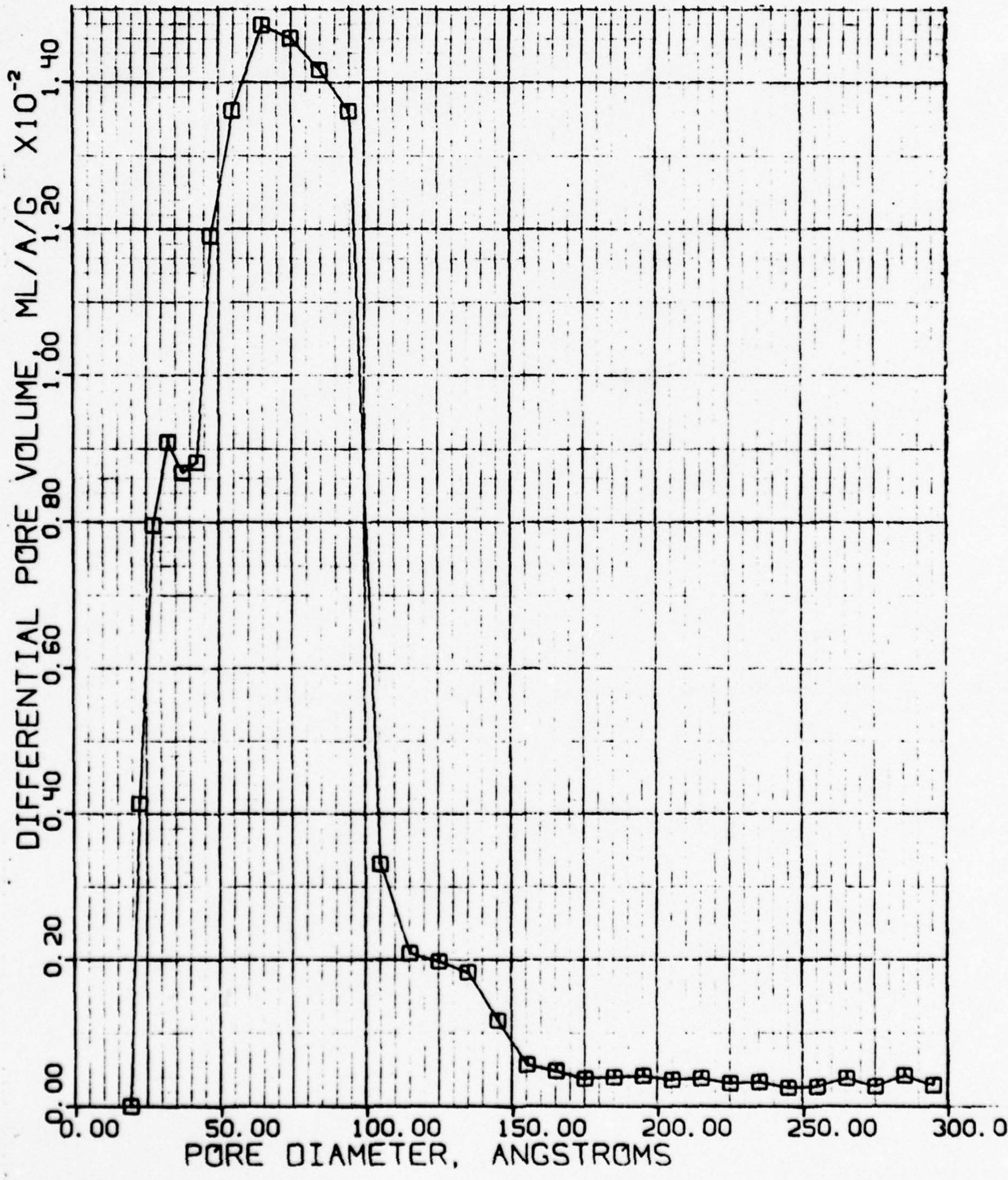


Figure 4

Ratio of Differential Pore Volume Distributions of Kocite Electrocatalyst 3289-127A and Kocite Material 3289-120

The histogram plots the ratio of the differential pore volume at each pore diameter. The dashed line indicates the expected ratio just considering the weight changes caused by leaching and impregnation with 17.3 wt-% platinum.

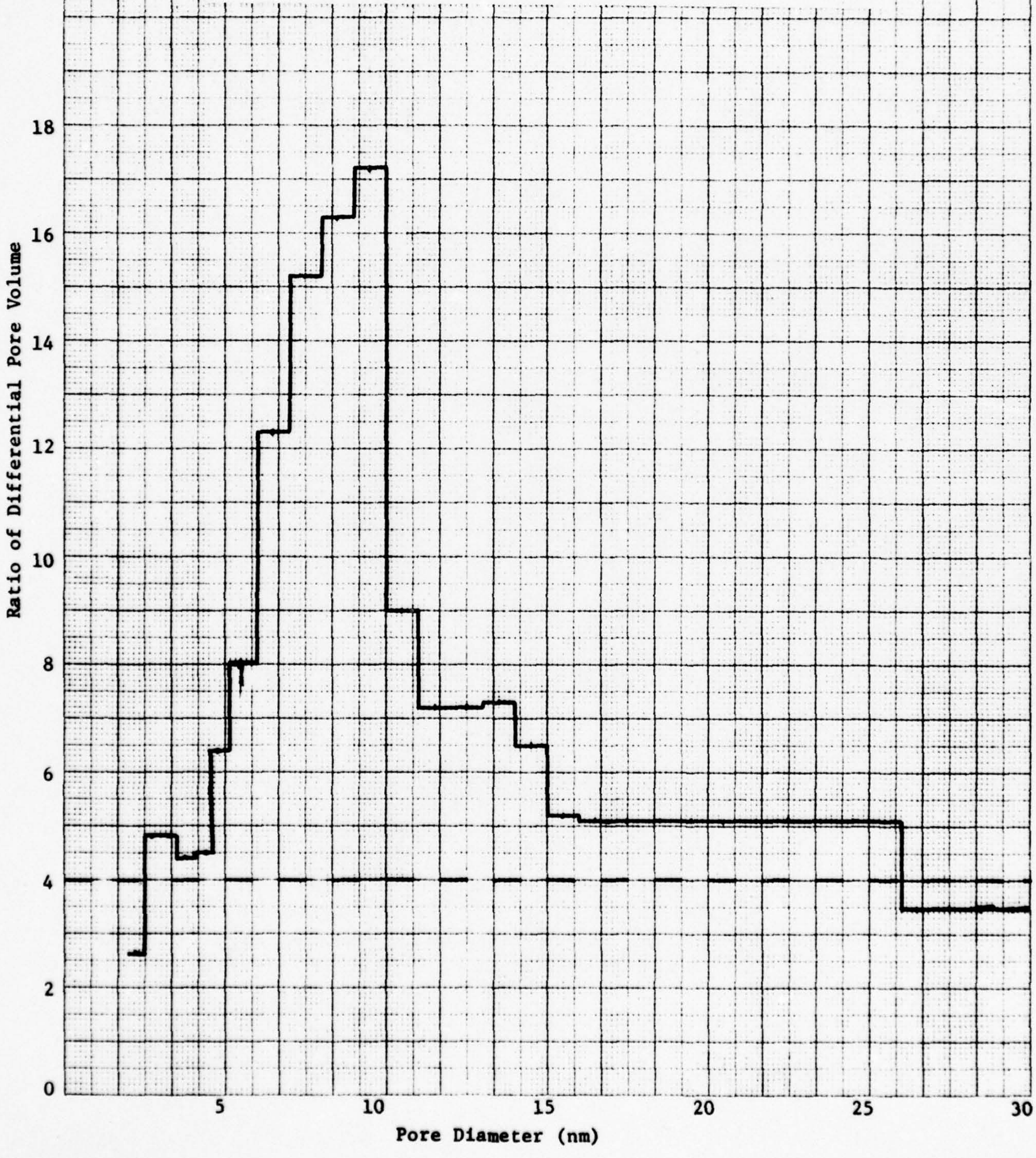


Figure 5

Scanning Electron Micrograph of
Kocite Electrocatalyst 3576-61B
on Electrode Surface

IV. ELECTRODE FABRICATION AND TESTING

4.1 Electrode Fabrication

During the last nine month period, ERC has prepared Kocite electrodes by two separate techniques, 1) the standard ERC machine calendering process used previously and described in earlier reports[2], and 2) an experimental process utilizing a sheet mold technique. Machine calendered (rolled) electrodes are fabricated by rolling a discrete catalyst layer and then pressure bonding this layer to a carbon fiber paper electrode substrate. In contrast, in the sheet mold process a catalyst-PTFE mixture is deposited directly onto the carbon fiber paper. The density of the sheet mold catalyst layer is equivalent to the medium density machine calendered catalyst layer used during the previous contract period.[2] Sintering conditions of both types of catalyst layers are similar to those described in previous reports.[2]

Teflon contents of both types of catalyst layers are typically between 40 and 50 weight percent. The lower Teflon content was emphasized in order to obtain peak electrode performance quickly at the risk of later electrode flooding. Electrode performance is expected to have superior long term stability at the higher Teflon loading, which is equivalent to the optimal Teflon loadings used during the previous contract period.[2]

4.2 Cell Testing

Details of cell testing techniques and apparatus have been discussed in earlier reports.[1,2] No changes in cell components have been made during the present period. All cells were operated at 180°C with an ERC supplied Kynol matrix and employed, with two exceptions, a 2 mg/cm^2 Pt black counter electrode. The other two cells were operated with Kocite electrodes as both cathode and anode.

During the previous contract period, the best cell performance was obtained with an experimental fibrous silicon carbide matrix prepared by ERC.[2] This matrix could not be used during the present contract period because of both reproducibility and materials availability problems. Other ERC silicon carbide matrix fabrication techniques were not judged by ERC to be sufficiently developed to warrant their use with Kocite electrodes.

The most important change in cell testing procedures made during the present contract period was the assembly of a number of cells at ERC where the Kocite electrode was not prewetted or float-filled in phosphoric acid. Deletion of this step was possible with Kocite electrocatalysts made from leached Kocite materials, in part, because of the improved electrocatalyst utilization characteristics ERC believes are obtainable with the sheet mold electrode.

V. RESULTS AND DISCUSSION

5.1 Introduction

In this section, the characteristics and performance of Kocite electrodes tested at UOP and ERC during the last nine month contract period are presented. Where possible, the cell performance is correlated with Kocite electrocatalyst and Kocite electrode properties. The results obtained during the last nine month period are summarized in section 5.3.

5.2 Cell Performance Results

The performance results of model phosphoric acid electrolyte fuel cells with a Kocite electrode used as the cathode are reported in this section. The characteristics of the Kocite electrodes and cell builds tested at ERC and UOP are listed in Tables VIII and IX respectively. A total of 47 cells were assembled and tested. The cell build characteristics and performance levels are reported only for those cells which reached the performance goals of the previous contract period, 0.6 volts at 100 ma/cm^2 and 180°C with a 0.5 mg/cm^2 Pt-loaded Kocite cathode. Over 50% of cells assembled reached this level. The remaining cells failed to reach this performance level either as a result of cross gas leaks, electrode flooding, cell hardware problems, or problems associated with the reproducibility of sheet mold electrodes.

Three Kocite electrocatalysts were tested in electrodes at ERC, 3289-127A, 3576-61A and 3576-61B. As indicated in Tables VI and VII, these electrocatalysts had the highest Pt dispersion achieved for the Pt-impregnation of leached Kocite materials by the respective Pt-impregnation techniques. Most of the cells tested used sheet mold Kocite electrodes with a 40 wt-% Teflon content as shown in Table VIII. Kocite electrode Pt-loadings were normally between 0.50 and 0.60 mg/cm^2 for the 2" x 2" electrodes. A substantial number of the cells tested at ERC were assembled with no Kocite electrode prewetting.

At UOP, cell testing activity was reduced from that of the previous contract period because of the emphasis on Kocite electrocatalyst production by various Pt-impregnation techniques. As a result, many of the Kocite electrode batches produced by ERC were tested only at ERC. Testing at UOP was primarily limited to electrodes from Kocite electrocatalyst batch 3289-127A. From this batch both machine calendered and sheet mold electrodes were tested. A few electrodes were tested from other electrocatalyst batches without notable success.

Summaries of full cell performance results at 180°C obtained at both ERC and UOP are presented in Tables X and XI. The most significant result presented in these tables is that for four cell builds, one of which is an all Kocite electrode cell, the peak full cell terminal voltage reached or exceeded the performance goal of 0.60 volts terminal at 200 ma/cm^2 set for this contract period.

Table VIII

Kocite^R Electrode Characteristics - ERC

Cell Build Number	Kocite Electrocatalyst Batch	Platinum Concentration $\frac{Pt}{Pt+C}$ (Wt-%)	Electrocatalyst Loading (mg/cm^2)	Platinum Loading (mg/cm^2)	PTFE Content (wt-%)	Fabrication Method	Electrode Prewetting (days)
ERC-113	3289-127A	17.3	3.2	0.55	50	Machine Rolled	1
ERC-117	3289-127A	17.3	3.8	0.65	50	Sheet Mold	None
ERC-119	3289-127A	17.3	3.8	0.65	50	Sheet Mold	1
ERC-122	3289-127A	17.3	3.26	0.56	40	Sheet Mold	3
ERC-123	3289-127A	17.3	3.26	0.56	40	Sheet Mold	3
ERC-125	3289-127A	17.3	3.26	0.56	40	Sheet Mold	3
ERC-126	3289-127A	17.3	3.33	0.58	40	Sheet Mold	1
ERC-127	3289-127A	17.3	3.33	0.58	40	Sheet Mold	1
ERC-132	3576-61B	16.3	3.46	0.56	40	Sheet Mold	0.1
ERC-133	3576-61B	16.3	3.46	0.56	40	Sheet Mold	None
ERC-134	3576-61A	15	3.33	0.50	40	Sheet Mold	None
ERC-136	3576-61B	16.3	3.46	0.56	40	Sheet Mold	None
ERC-138	3576-61B	16.3	3.66	0.60	40	Sheet Mold	None
ERC-139*	3576-61B	16.3	3.66	0.60	40	Sheet Mold	None
ERC-140*	3576-61B	16.3	3.66	0.60	40	Sheet Mold	None

*Kocite electrodes used as cathode and anode.

Table IX

Kocite^{R} Electrode Characteristics - UOP

Cell Build Number	Platinum Concentration $\frac{Pt}{PttC}$ (wt-%)	Electrocatalyst Loading (mg/cm^2)	Platinum Loading (mg/cm^2)	PTFE Content (wt-%)	Fabrication Method	Electrode Prewetting (days)
3289-127A-1	17.3	3.33	0.58	50	Machine Rolled	None
3289-127A-2	17.3	3.33	0.58	50	Machine Rolled	None
3289-127A-3	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-4	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-5	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-6	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-7	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-8	17.3	3.33	0.58	50	Machine Rolled	1
3289-127A-13	17.3	2.72	0.47	50	Sheet Mold	4
3289-127A-14	17.3	2.72	0.47	50	Sheet Mold	4

Table X

Summary of ERC Cell Performance Results at 180°C

Cell Build Number	Kocite Electrocatalyst Batch	Platinum Loading (mg/cm^2)	Terminal Voltage[a] (V) @ 100 ma/cm^2		Terminal Voltage[a] (V) @ 200 ma/cm^2		Days Tested	Peak Performance	Comments
			Air	Oxygen	Air	Oxygen			
ERC-113	3289-127A	0.55	0.670	0.725	0.600	0.665	181	73 days	Stable
ERC-117	3289-127A	0.65	0.610	0.680			1	1 day	Cross gas leak
ERC-119	3289-127A	0.65	0.600	0.660			7	6 days	Stable
ERC-122	3289-127A	0.56	0.650	0.710	0.580	0.655	9	6 days	Slight decay
ERC-123	3289-127A	0.56	0.610	0.680			5	2 days	Stable
ERC-125	3289-127A	0.56	0.640	0.700	0.570	0.635	9	5 days	Decayed
ERC-126	3289-127A	0.58	0.620	0.700			1	1 day	
ERC-127	3289-127A	0.58	0.620	0.690			1	1 day	
ERC-132	3576-61B	0.56	0.670	0.730	0.600	0.670	71	1 day	Not stable
ERC-133	3576-61B	0.56	0.665	0.720	0.590	0.660	64	1 day	Not stable
ERC-134	3576-61A	0.50	0.600	0.680			4	1 day	Not stable
ERC-136	3576-61B	0.56	0.600	0.670			64	2 days	Stable
ERC-138	3576-61B	0.60	0.680	0.740	0.620	0.685	51	22 days	Stable
ERC-139[b]	3576-61B	0.60	0.625	0.695	0.550	0.625	37	3 days	Slight cross gas leak
ERC-140[b]	3576-61B	0.60	0.670	0.730	0.600	0.670	7	7 days	Stable

[a] Cell voltage obtained with Kocite cathode and 2 mg/cm^2 Pt-black counter electrode; cell voltage measured at current collector plates.

[b] Kocite electrodes used as cathode and anode.

Table XI

Summary of UOP Cell Performance Results at 180°C

Cell Build Number	Platinum Loading (mg/cm^2)	Terminal Voltage[a] (V) @ 100 ma/cm^2		Terminal Voltage[a] (V) @ 200 ma/cm^2		Days Tested	Peak Performance	Comments
		Air	Oxygen	Air	Oxygen			
3289-127A-1	0.58	0.604	0.674	0.518	0.599	10	3 days	Gas crossover
3289-127A-2	0.58	0.600	0.672	0.532	0.606	7	3 days	Gas crossover
3289-127A-3	0.58	0.620	0.692	0.547	0.631	9	2 days	Gas crossover
3289-127A-4	0.58	0.610	0.681	0.532	0.611	9	2 days	Gas crossover
3289-127A-5	0.58	0.629	0.687	0.548	0.635	37	31 days	Gas crossover
3289-127A-6	0.58	0.650	0.716	0.585	0.666	72	28 days	Reverse polarized
3289-127A-7	0.58	0.622	0.669	0.545	0.622	14	8 days	Gas crossover
3289-127A-8	0.58	0.618	0.674	0.540	0.607	23	18 days	Decayed
3289-127A-13	0.47	0.618	0.702	0.541	0.631	36	3 days	Reverse polarized
3289-127A-14	0.47	0.644	0.712	0.556	0.641	72	52 days	Continuing with decay

[a] Cell voltage obtained with Kocite cathode and 2 mg/cm^2 Pt-black counter electrode; cell voltage measured at current collector plates.

Two of these cells, ERC-113 and ERC-128, will be discussed in more detail later in this section. Comparing the results of cell ERC 138 with the best results obtained during the previous contract period, listed in Table II, shows that for cells with a Kynol matrix, cell performance improvements of 30 mV at 100 ma/cm^2 and 55 mV at 200 ma/cm^2 were obtained.

Inspection of Tables X and XI indicates that low oxygen gain figures are obtainable with Kocite electrodes, which is indicative of low diffusion losses. The average O_2 gain at 200 ma/cm^2 for the three cells achieving the performance goal is only 67 mV.

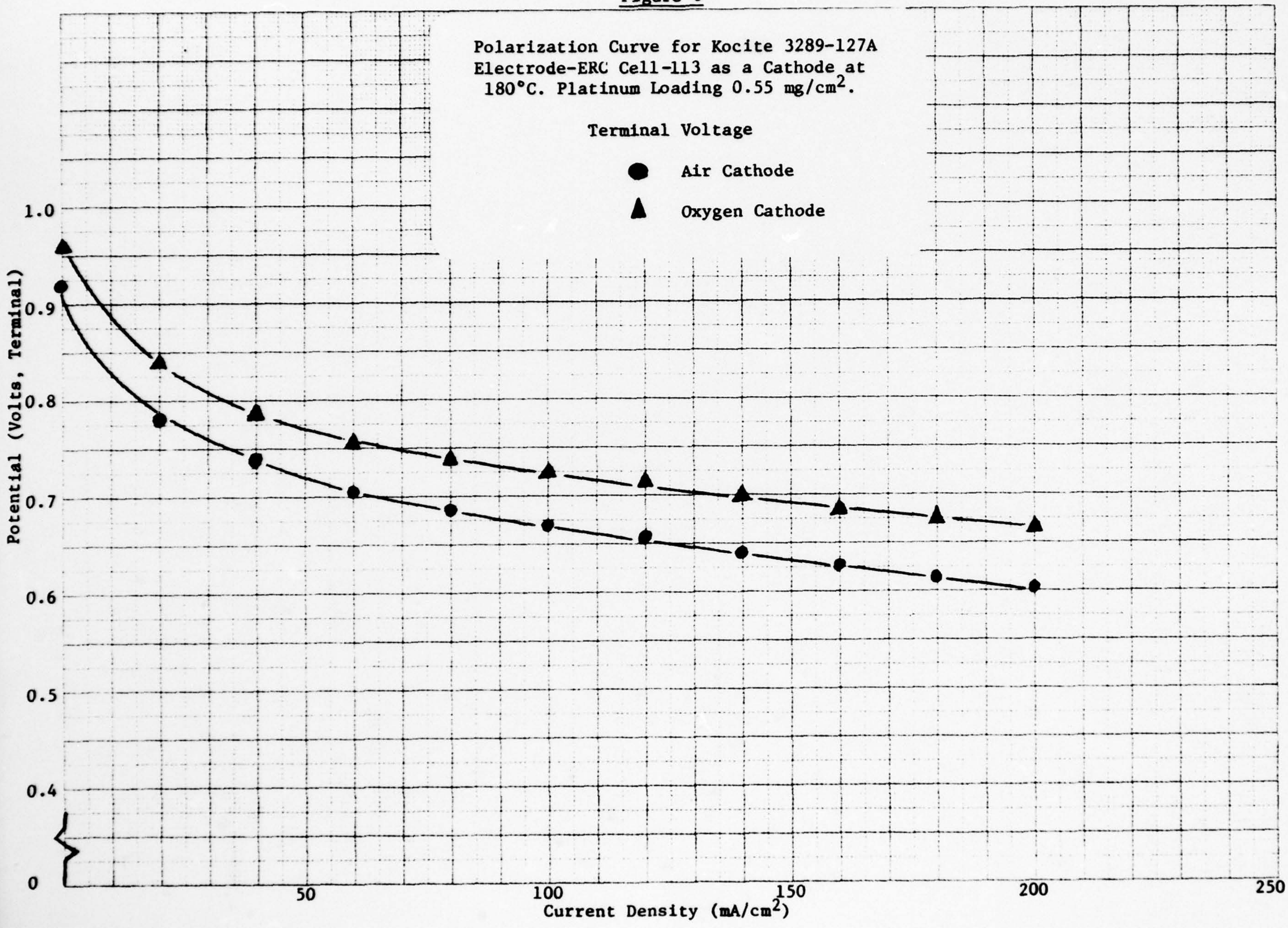
The current contract performance goal has been met with Kocite electrodes fabricated from both Kocite electrocatalyst batches 3289-127A and 3289-61B with both machine calendered and sheet mold electrodes. Cells containing Kocite electrodes with 50 wt-% Teflon contents generally did not reach peak performance until several days after startup. In the case of cell ERC-113 the peak cell performance was not reached until the 73rd day. However, at 40 wt-% Teflon content, used for many of the later Kocite electrodes, peak performance was normally reached much sooner. At this Teflon content many, but not all, cells showed electrode flooding problems so endurance testing was generally not performed. From the results indicated in Tables X and XI, a Kocite electrode Teflon composition of 50 wt-% appears to be desirable for long term electrode stability.

Cells assembled using Kocite electrodes fabricated from electrocatalyst batch 3576-61A, prepared by the PDD impregnation technique, did not perform as well as expected based on the X-ray diffraction determination of Pt crystallite size (shown in Table VII). While the testing of this electrocatalyst was not extensive, only one cell, ERC-134, reached 0.60 volts at 100 ma/cm^2. The reasons for the relatively poor performance of electrodes fabricated from this electrocatalyst batch are not presently understood.

The best cell performance reached at UOP was on cell 3289-127A-6, which was stable until it was accidentally reversed polarized by the power supply driving it at 200 ma/cm^2. A large number of cells developed gas crossover problems early during cell testing and were subsequently terminated. It is speculated that this problem arose from the use of a particular Kynol matrix batch. With the limited testing performed at UOP, no cell reached the specified performance goals. The results obtained at ERC were not reproduced at UOP.

Both cells ERC-113 and ERC-138, which reached the performance goal, have been under test for more than 500 hrs., as indicated in Table X. To further characterize these cells, the oxygen and air polarization curve of cell ERC-113 at its performance peak and its performance as a function of time are shown in Figures 6 and 7, respectively. Similar curves are shown for cell ERC-138 in Figures 8 and 9. In the case of cell ERC-138, the polarization curves were extended to 250 ma/cm^2. At this current a terminal voltage of 0.595

Figure 6



Polarization Curve for Kocite 3289-127A Electrode-ERC Cell-113 as a Cathode at 180°C. Platinum Loading 0.55 mg/cm^2.

Figure 7

Lifetime Test for Kocite 3289-127A
Electrode-ERC Cell 113 as a Cathode at
180°C. Operated Normally at 100 ma/cm^2.
Platinum Loading is 0.55 mg/cm^2.

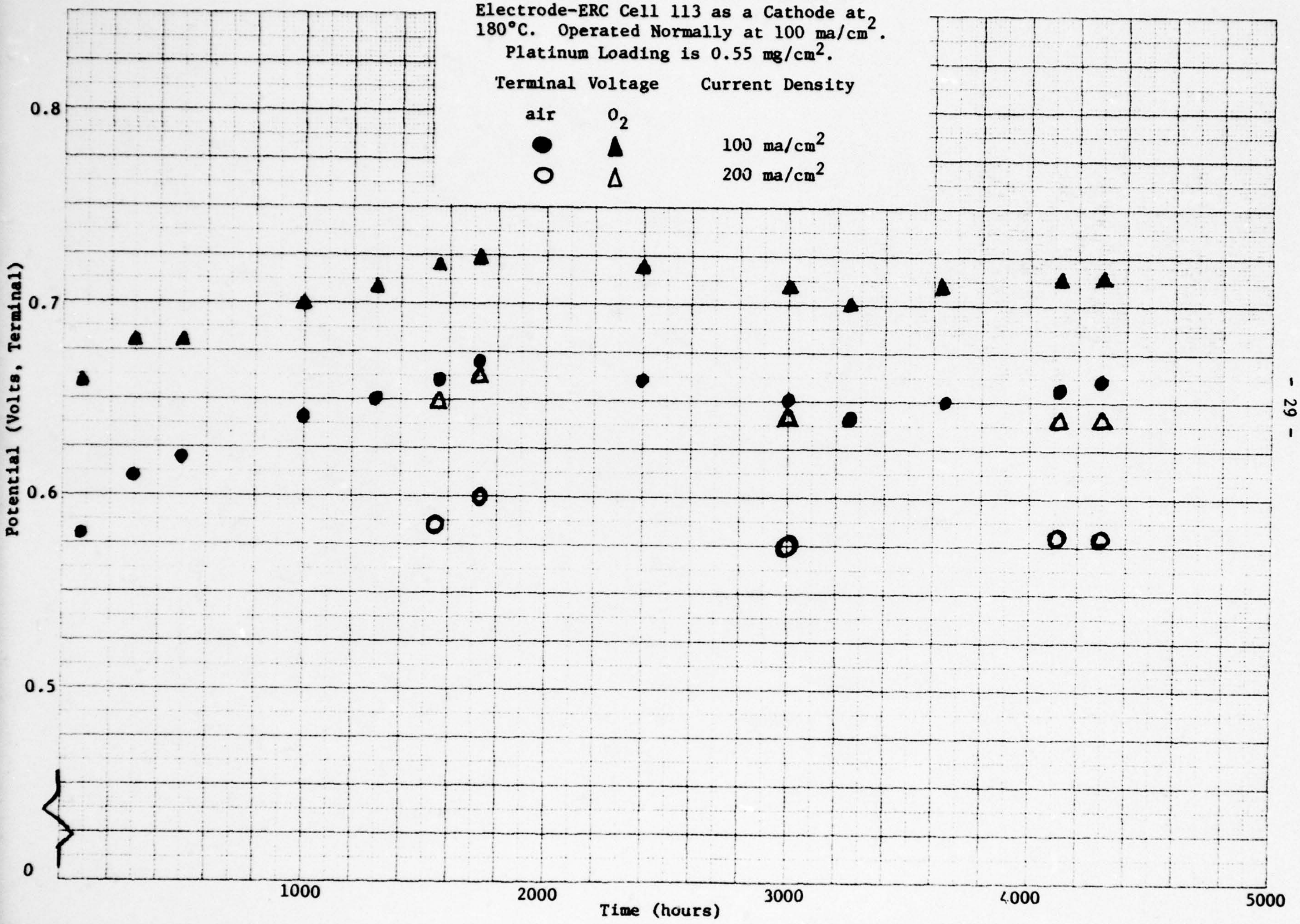
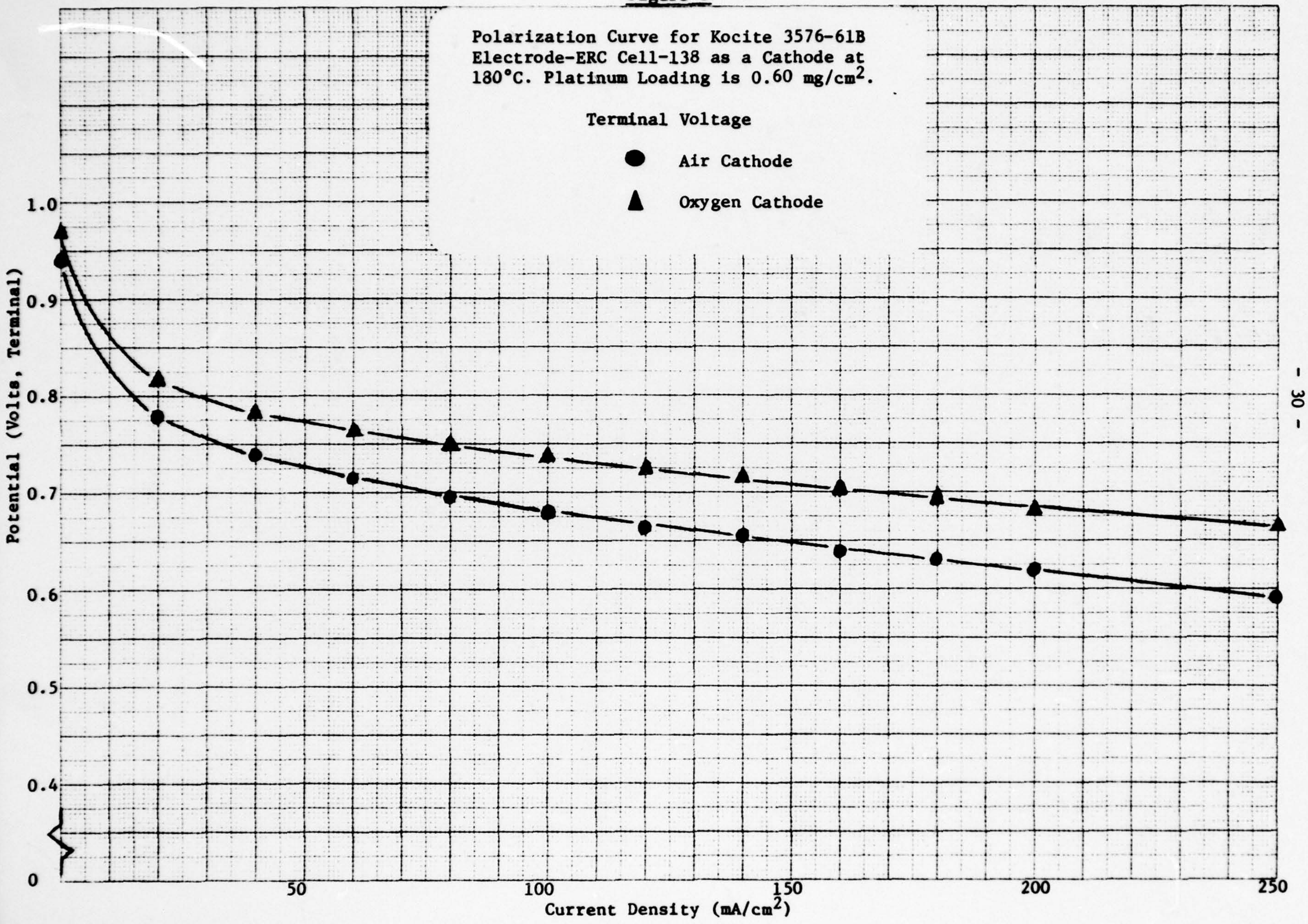


Figure 8



Polarization Curve for Kocite 3576-61B Electrode-ERC Cell-138 as a Cathode at 180°C. Platinum Loading is 0.60 mg/cm^2.

volts on air was achieved. As shown in Figures 7 and 9, both cells have good long term stability with cell ERC-113 operating more than 4000 hours and cell ERC-138 more than 1250 hours. The data in these figures emphasize the low oxygen gain figures of these cells, with cell ERC-138 having an oxygen gain of only 75 mV at 250 ma/cm^2.

To determine if improvements in cell performance might be obtained by using a SiC matrix rather than a Kynol matrix, ERC has analyzed the various contributions to the polarization losses observed in increasing the cell current from 100 to 200 ma/cm^2. Activation polarization losses are estimated to be 27 mV assuming a Tafel slope of 90 mV per decade. Ionic polarization losses, caused mainly by the resistance in the cell matrix, are estimated to be in the range of 30 to 40 mV for each 100 ma/cm^2 with the presently used Kynol matrix. Diffusion polarization losses on air are believed to be low at 100 ma/cm^2 with properly wet-proofed Kocite electrodes having small catalyst particle sizes, but probably are a factor at higher current densities. A diffusion loss of 5 mV from 100 to 200 ma/cm^2 is thought by ERC to be a reasonable estimate for a good Kocite electrode. Thus a doubling of the cell current from 100 to 200 ma/cm^2 should give a minimum polarization loss of 60 to 70 mV assuming that anode polarization losses may be neglected. As indicated in Table II, the best cell performance results obtained during the previous contract period exceed this estimate by 20 to 30 mV indicating somewhat larger diffusion losses. Inspection of the cell performance results listed in Tables X and XI shows that for many of the best cells tested during the present contract period, the polarization loss is equal to the estimated polarization loss for a good electrode. This suggests that diffusion losses are quite small and further improvement in cell performance will require improved Pt utilization in the Kocite electrode and a lower ionic resistance of the cell matrix.

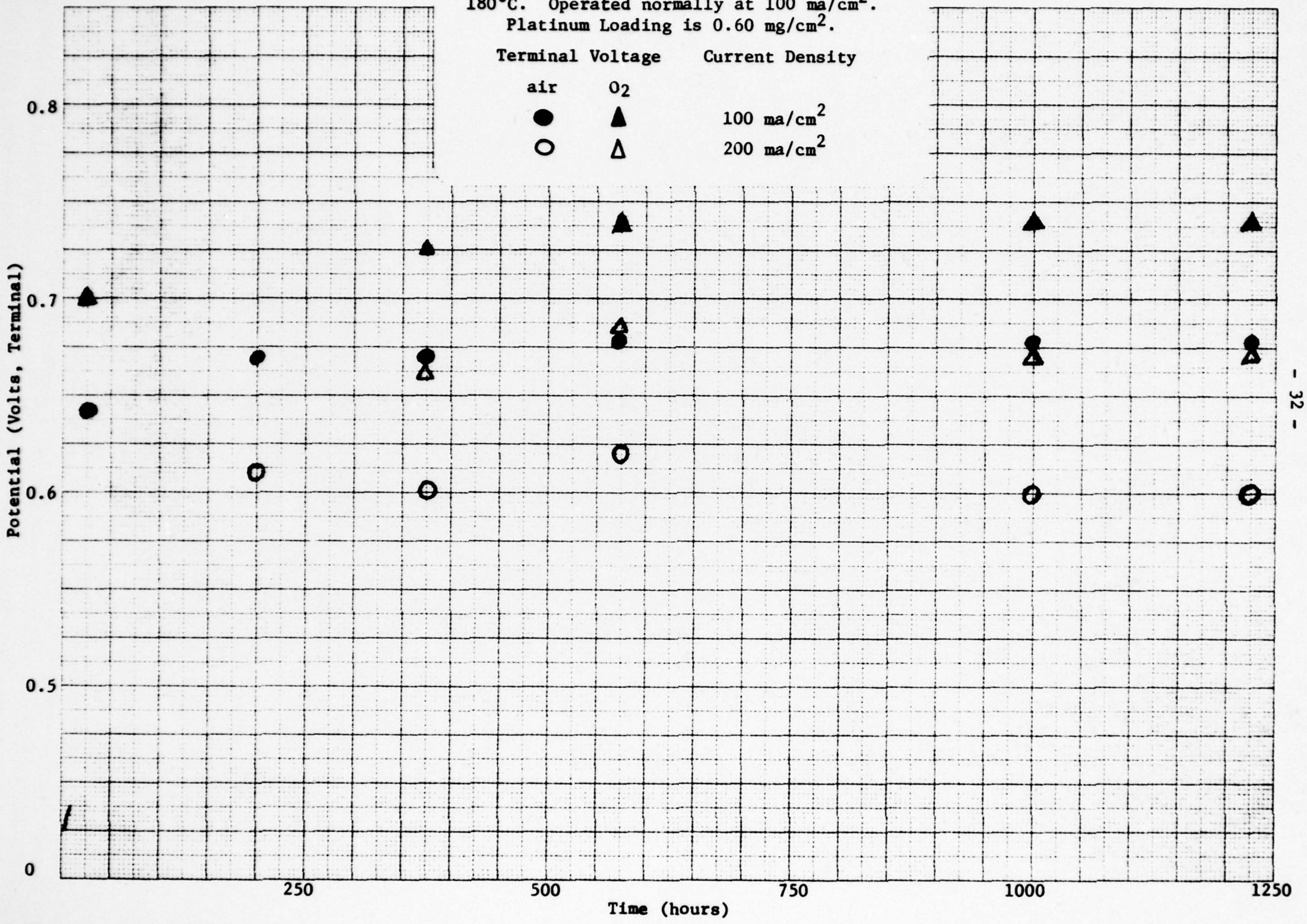
5.3 Summary of Results

The most important results obtained during this contract period are summarized below.

(A) Production of Leached Kocite Materials

A variety of leaching conditions have been investigated to determine the preferred conditions under which the alumina substrate is leached from Kocite particles by H_3PO_4 to produce a high-surface-area pyropolymer structure serving as the electrically conductive support for highly dispersed Pt crystallites. Depending on the initial substrate surface area and carbon content of the Kocite particles, surface areas up to 1000 m^2/g can now be routinely produced. To accomplish this, Kocite particles are leached by the H_3PO_4 for 24 hours at 140°C.

Figure 9



Lifetime Test for Kocite 3576-61B
Electrode-ERC Cell 138 as a Cathode at
180°C. Operated normally at 100 ma/cm^2.
Platinum Loading is 0.60 mg/cm^2.

(B) Kocite Electrocatalyst Production

i. Impregnation of Leached Kocite Materials

Kocite electrocatalysts have been produced by the Pt impregnation of leached Kocite materials using chloroplatinic acid, platinum diaminedinitrite, and platinum acetylacetonate. The highest Pt dispersions were obtained using platinum diaminedinitrite, although, the best fuel cell performance was obtained using electrocatalysts prepared by chloroplatinic acid impregnation.

ii. Kocite Electrocatalyst Structure

Initial efforts directed at determining Kocite electrocatalyst pore structure and particle shape were made using pore volume distribution measurements and high resolution Scanning Electron Microscopy. These results indicate that

a. Kocite structure is not destroyed by either leaching the substrate from the Kocite particles nor the catalyzation process.

b. Currently used Kocite electrocatalysts have a platelet shape.

c. The maximum in the pore volume distribution is shifted from pore diameters of $\sim$4.0 nm for a typical Kocite material to $\sim$7.0 nm for the resultant electrocatalyst with the substrate materials and process conditions employed currently.

(C) Cell Performance

i. Cell Performance Level

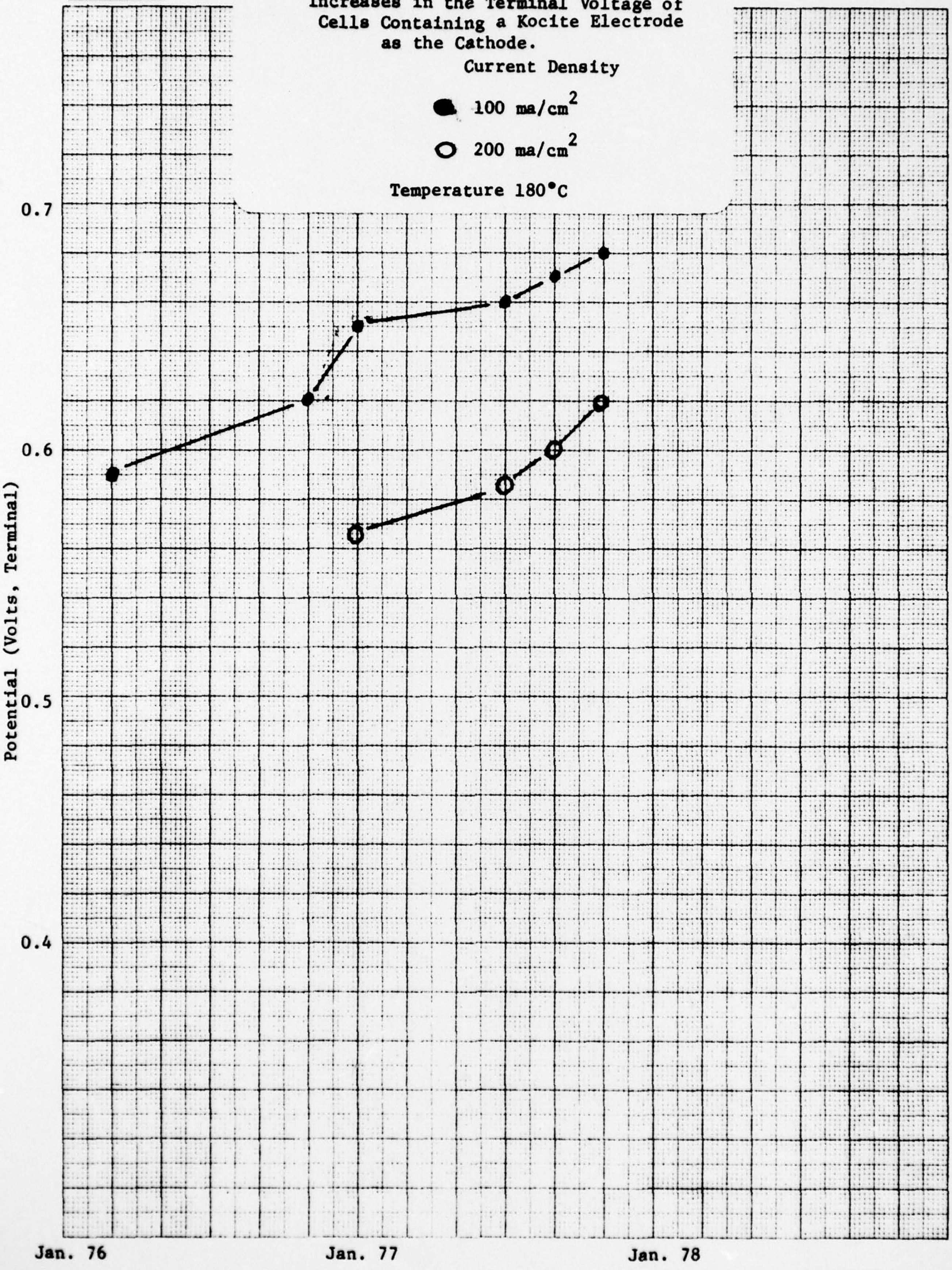
Cells with Kocite cathodes fabricated from different Kocite electrocatalysts and by both machine calendering and sheet mold techniques have reached or exceeded the contract performance goals of 0.60 volts at 200 ma/cm^2. These cells were operated at 180°C with $\sim$0.5 mg/cm^2 Pt-loaded Kocite electrodes and used a Kynol matrix. The maximum full cell performance achieved was 0.62 volts at 200 ma/cm^2 with an oxygen gain at this current of 65 mV. The improvement in cell performance during the current and previous contract periods is shown in Figure 10. Of two cells constructed with Kocite electrodes as both anode and cathode, one reached the contract performance goal with no prewetting of either electrode.

ii. Endurance Testing

Endurance testing on cells achieving the performance goal were performed. The longest running cell has operated stably in excess of 4000 hours.

Figure 10

Increases in the Terminal Voltage of Cells Containing a Kocite Electrode as the Cathode.



iii. Electrode Structure

The best results of this contract period were achieved with a sheet mold electrode having a 40 wt-% Teflon content. Results with other cells indicate that an electrode with 50 wt-% Teflon should have improved long term stability by being more resistance to electrode flooding.

iv. Electrode Prewetting

Cells assembled with sheet mold electrodes, with no electrode prewetting, achieved the best performance during this contract period.

VI. CONCLUSIONS

Based on the results obtained in the last nine months in the areas of Kocite particle substrate leaching, Kocite electrocatalyst preparation, improved Kocite electrode performance, and new electrode structures, the following conclusions concerning low cost fuel cell electrodes fabricated from Kocite electrocatalysts are warranted.

(A) Leached Kocite materials can be routinely prepared to produce high-surface-area ($\lesssim 1000$ m^2/g) electrocatalyst supports with a narrow pore volume distribution. The structure appears to be stable both through the leaching and catalyzation process.

(B) The best Kocite electrocatalysts prepared during this contract period were made by Pt-impregnation of leached Kocite material with chloroplatinic acid.

(C) Full cell performance levels of 0.68 V at 100 ma/cm^2 and 0.62 V at 200 ma/cm^2 can be achieved by cells with a ~ 0.5 mg/cm^2 Pt-loaded Kocite cathode operated at 180°C with a Kynol matrix.

(D) Optimal fuel cell performance can be obtained without prewetting the Kocite electrode with H_3PO_4.

(E) Improvements in cell performance may be achieved by

- i. Achieving and maintaining improved Pt dispersions.
- ii. Producing the electrocatalyst surface chemistry and pore volume distribution to maintain the optimum three-phase boundry.
- iii. Increasing Pt utilization with improved electrode structures.
- iv. Using a matrix (probably SiC) with reduced ionic resistance.

(F) Substantially increased efforts must be made in cell testing to establish cell reliability and reproducibility at the performance levels attained during the present contract period.

(G) Variations in performance between cells observed during the present contract period are believed to arise from differences in the Kocite electrocatalysts with different impregnation techniques, lack of optimization of electrode structure for leached Kocite electrocatalysts, and the experimental nature of the sheet mold electrodes. Further experimentation in these areas should lead to more consistent cell performance levels.

VII. RECOMMENDATIONS

To further improve the performance of Kocite electrocatalysts and Kocite electrodes, and continue their development in H_3PO_4 electrolyte fuel cells, the following recommendations are made.

(A) Continued efforts must be made to achieve higher Pt dispersions (smaller Pt crystallite size) on leached Kocite supports.

(B) The structural analysis of Kocite electrocatalysts should be continued to determine the effect of process conditions and impregnation technique on both pore and particle structures.

(C) The structural parameters of sheet mold Kocite electrodes should be varied and optimized to obtain increased Pt utilization and reliable wet proofing. To allow this, 50 g electrocatalyst batch should normally be produced.

(D) Kocite electrodes should be tested in sufficient quantities and with uniform cell hardware and matrix material to establish electrode reliability and reproducibility.

(E) Extended lifetime testing (5000 hrs. or more) of Kocite electrodes should be expanded.

VIII. REFERENCES

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3. Pictures taken at Scanning Electron Microscope Users' Laboratory at the University of Chicago. The lab is operated on a resource grant from the National Institutes of Health.

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