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ADVANCED WATER-COOLED PHOSPHORIC ACID

FUEL CELL DEVELOPMENT

TECHNICAL PROGRESS REPORT NO. 14

A QUARTERLY REPORT
for
APRIL - JUNE 1989

Contract DE-AC21-88MC24221

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Prepared for

U.S. Department of Energy
Morgantown Energy Technology Center
P.O. Box 880, 3610 Collins Ferry Road
Morgantown, WV 26505-0880

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P.O. Box 739
195 Governors Highway
South Windsor, Connecticut 06074

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PROGRAM HIGHLIGHTS

- Electrode substrate handsheets were formed meeting surface finish thickness and density requirements. A forming trial for full-size substrates has been scheduled.
- A new electrode edge seal with in-plane bubble pressures of 40-50 psid and through-plane bubble pressures of 8-9 psid was demonstrated.
- A new polymeric edge seal for ERP's with bubble pressure greater than 30 psid was tested and shown to be stable after 5 thermal cycles.
- A thin (1.2-mil) matrix was applied to full-size electrodes using a curtain coater.
- Full-size coolers were fabricated using both molded and commercial graphite holders.

I. INTRODUCTION

The Advanced Water Cooled Phosphoric Acid Fuel Cell Development program is being conducted by International Fuel Cells Corporation (IFC) to improve the performance and minimize the cost of water cooled, electric utility phosphoric acid fuel cell stacks.

The program adapts the existing on-site Configuration B cell design to electric utility operating conditions and introduces additional new design features. Task 1 consists of the conceptual design of a full-scale electric utility cell stack that meets program objectives. Tasks 2 and 3 develop the materials and processes required to fabricate the components that meet the program objective. The design of the small area and three 10-ft^2 short stacks is conducted in Task 4. The conceptual design also is updated to incorporate the results of material and process developments, as well as results of stack tests conducted in Task 6. Fabrication and assembly of the short stacks is conducted in Task 5 and subsequent tests are conducted in Task 6. The Contractor expects to enter into a contract with The Electric Power Research Institute (EPRI) to assemble and endurance test the third 10-ft^2 short stack. The management and reporting functions of Task 7 provide DOE/METC with program visibility through required documentation and program reviews.

This report describes the cell design and development effort that is being conducted to demonstrate by subscale stack test, the technical achievements made toward the above program objectives.

II. STATUS

TASK I - CONCEPTUAL DESIGN

Objectives

The objective of this task is to define the conceptual design for a cell stack having a performance level of at least 175-W/ft2 and a manufactured cost of less than $400/kW. The stack must be capable of operating for a minimum of 40,000 hours at electric utility conditions.

Activity

This activity has been completed.

TASK 2 - COMPONENT DEVELOPMENT

Subtask 2.1 - Materials Development

Objectives

The objectives of this subtask are to provide an improved substrate, verify the stability of low cost substrate and electrolyte reservoir materials at pressurized cell operating conditions, and increase the thermal conductivity of the electrolyte reservoir.

Activity

Graphitized handsheets from the second precursor formation trial were characterized to determine their physical properties. The measured properties, with the exception of the electrical resistance, met requirements as shown in Table 2.1-1.

Based on the results of this handsheet trial, target values of density and thickness were selected for a full-size precursor forming trial.

Table 2.1-1

Thin Substrate Precursor Handsheet - Trial 2

	Target Value	Sample Number A3	B3	C3	A5	B5	C5
Density, GM/CC	.35 - .50	.39	.37	.41	.38	.40	.36
Comp. Strength, Psi	100 Min	104	129	117	103	123	—
Flex, Strength, Psi	300 Min	1247	1348	922	1077	1297	976
Elec. Resistance, mV/Mil	.025 Max	.085	.081	.080	.083	—	—
Corrosion Potential, mV	1150 Min	1269	1240	1224	1250	1238	1241

Subtask 2.2 - Single Cell Testing

Objectives

The objectives of this subtask are to test subscale cells to evaluate improvements in cell components consisting of substrates, electrolyte reservoir plates, catalyst and matrix; verify performance and endurance of components fabricated for short stacks; optimize the hydrophobicity of catalyst layers made for operation at power densities of 200 WSF or greater; evaluate a higher performing laboratory catalyst for this application, and define the permissible range of operating conditions.

Activity

Subscale cells tested during the month are listed in Table 2.2-1.

Cell 4315. This cell which was run to evaluate anode sinter temperature and cell pinch, completed 1600 hours with performance of 0.602V at 300 ASF, 14.7 psia and 415°F. The cell's performance history is shown in Figure 2.2-1.

Cells 4320 and 4321 were started as an initial trial with most electrolyte stored on the anode side of the cell. The cells were shut down after completing 1000 hours. Results were inconclusive and more testing is required to assess this configuration.

Table 2.2.1. Subscale Cells Tested During June 1989

Cell #	Anode	Cathode	Test Purpose
4315	0.27 mg HYCAN -sintered at 640°F	0.53 mg GSB-18	Effect of anode sinter temp. and cell "pinch" on performance
4320	0.28 mg HYCAN	0.54 mg GSB-18	To test cells with acid stored on the anode side
4321	0.28 mg HYCAN	0.54 mg GSB-18	
4327	0.28 mg HYCAN	0.49 mg GSB-18	
4334	0.25 mg HYCAN	0.50 mg GSB-18	To evaluate catalyst layers with improved wettability control
4335	0.25 mg HYCAN	0.50 mg GSB-18	

Cell 4327 continued the investigation of anode-side acid storage by wetproofing the cathode ERP. The superiority of this configuration is apparent when the performances of the three cells are compared. (Table 2.2-2.)

Table 2.2-2. Effect of Anode-side Electrolyte Storage

Cell No.	Hours	Cell Voltage at 300 ASF
4320	950	0.588
4321	928	0.562
4327	902	0.621

Performance history to date is shown in Figure 2.2-2. Cell 4327 will continue on test.

Two new cells incorporating anode and cathode catalyst layers with improved wettability control features were started this month as rig builds 4334 and 4335.

The following table shows that cells were able to run up to 400 ASF and then return to 300 ASF without any performance penalty.

Table 2.2-3. High Current Density Performance Effects

Cell No.	Volts at 200 ASF (hr)	Volts at 300 ASF (hr)	Volts at 400 ASF (hr)	Volts at 300 ASF (hr)
4334	0.67 (140)	0.624 (164)	0.586 (287)	0.628 (356)
4335	0.673 (136)	0.624 (160)	0.577 (283)	0.626 (352)

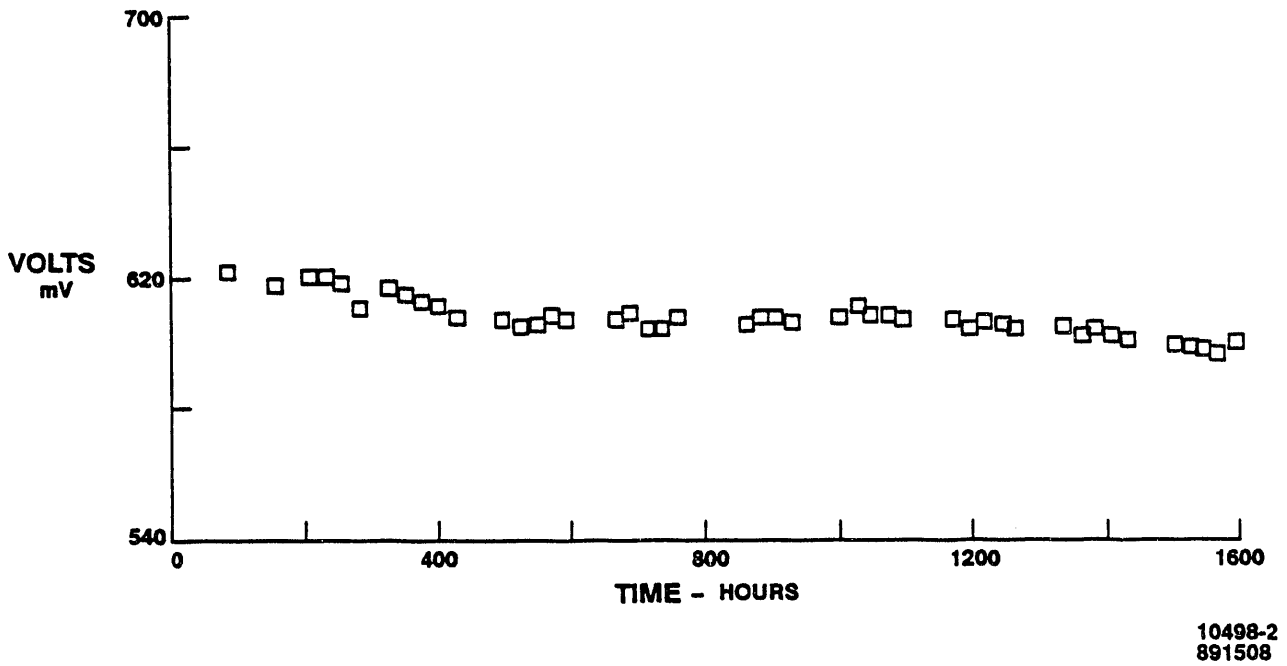

Figure 2.2-1. Performance History for Cell 4315 at 14.7 psia, 300 ASF/415°F

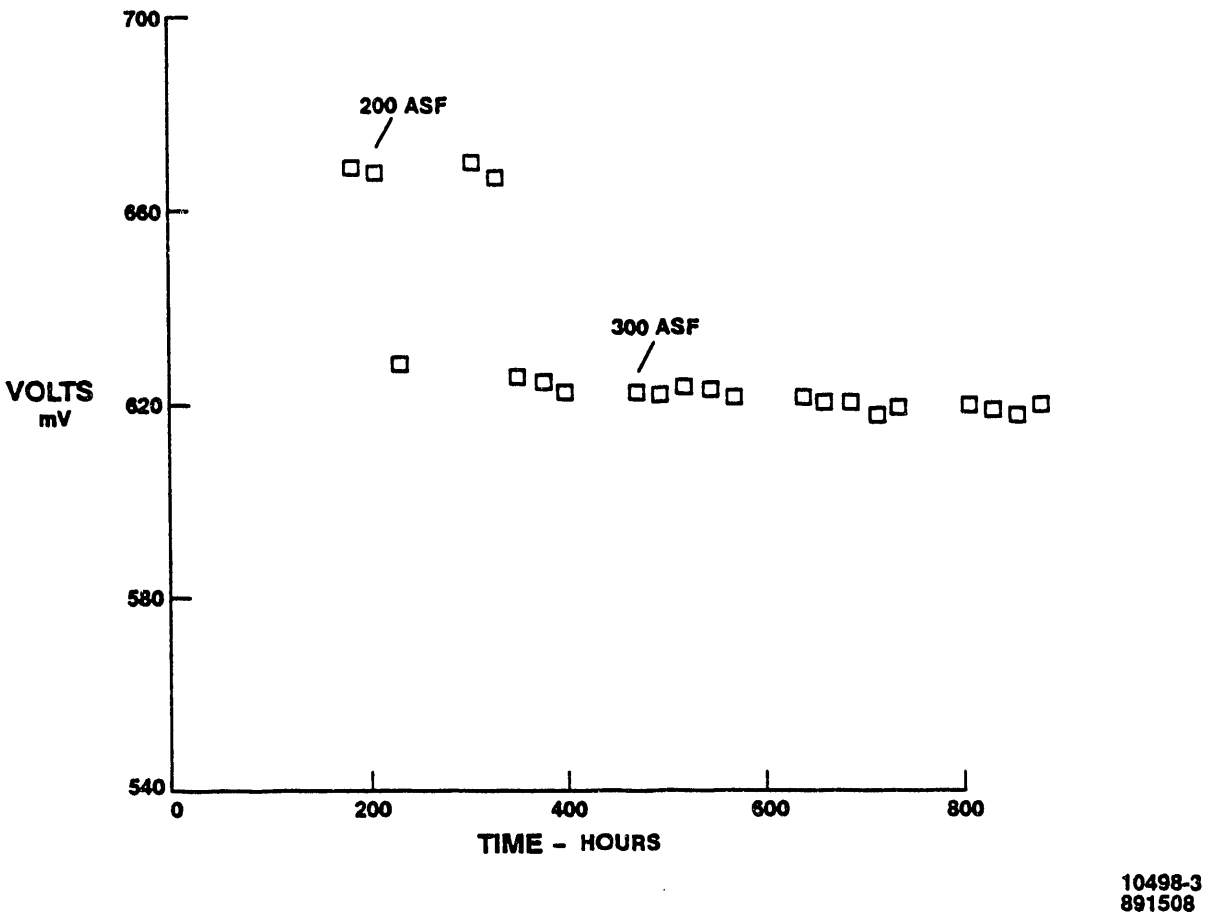

Figure 2.2-2. Performance History for Cell 4327 at 14.7 psia, 200 ASF/400°F and 300 ASF/415°F

TASK 3 - PROCESS DEVELOPMENT

Subtask 3.1 - Substrates

Objectives

The objectives of this subtask are to develop the process technologies required to produce electrode substrates and electrolyte reservoir plates that meet the technical requirements and cost objectives established in the conceptual design.

Activity

Target values of density and thickness have been selected for a full-size substrate precursor trial as reported in Subtask 2.1.

Material has been received for fabricating full-size electrolyte reservoir plates on the double belt press.

Subtask 3.2 - Integral Separator

Objectives

The objective of this subtask is to develop the process technology to fabricate integral separators, up to 10-ft2 in size, that meet the technical requirements and cost objectives established in the conceptual design.

Activity

Samples of the skived sheet material formed by a segmented molding process were submitted for laboratory analysis of the segment bond joint composition; photomicrographs of material cross sections show discontinuous composition at the joint zone. Subscale test samples of integral separator plates, using this new skived material, have been prepared and will be evaluated against similar plates formed with non-segment molded skived material.

Both new and used ISP samples are being tested to establish present baseline performance and to quantify alternate configuration performance improvements. The objective is to obtain a relative comparison of the effective plate barrier against acid or gaseous transfer between cells.

Subtask 3.3 - Seals and Matrix

Objectives

The objective of this subtask is to develop the technology to fabricate higher integrity seals and matrices that meet the technical requirements and cost objectives established in the conceptual design.

Activity

Fillerband

Work on forming edge seals, fillerband and matrix simultaneously continues. The focus this quarter has been on forming a smooth fillerband with a high bubble pressure. The baseline fillerband was measured to have only 1/2 psid cross pressure bubble pressure alone and 2-3 psid with a thin matrix applied. The best results of the current effort is 14-15 psid. The following table compares the baseline fillerband with the experimental fillerband.

Table 3.3-1. Fillerband Ink Comparison

	Baseline	Experimental
Wt% solids	45%	68%
Thru-Plane Bubble Pressure	2-3 Psid	8-14 Psid

Four inch by four inch electrode samples that are catalyzed over ~50% of the area are being used to demonstrate the feasibility of the combined process. A precoat operation has been added to the fillerband area to improve the surface uniformity of the matrix in this area. Bubble pressures measured on the edge seal and fillerband of samples made in one operation are not as high as when the edge seal and fillerband were formed separately with the same ink formulations. The details of impregnating and coating the 4" x 4" samples are unique and interfere with getting the desired result. We will increase our sample size to about 12" x 12" to avoid some of the problems associated with smaller samples.

Electrode/ERP Edge Seals

Work on an alternate polymeric seal concept continues. Polymer seals differ from impregnated seals in three ways as shown in Table 3.3–2.

Table 3.3–2. Seal Concepts

	Impregnated	Polymer
Material	Submicron Carbon/Graphite	Fluorcarbon Polymer
Processing	Hydraulic Intrusion of Aqueous Ink	Roll Coating of Solution
Functional	Wet Seal	Impervious Coating

Photomicrographs show that initial ERP polymer edge seal samples were only partially filled with polymer. The surface build up of polymer appears to provide the seal. One group of samples was made with a single coat of polymeric solution applied with a brush, the second set had two coats. The bubble pressure of the edge seals was over 30 psid. Bubble pressures remained over 30 psid through 5 thermal cycles from room temperature to 400°F. The samples will be thermal cycled at least 10 times to see if the bubble pressure degrades. Effort is being focused on a practical production process to integrate the polymeric seal into ERP's.

Matrices

A curtain coater trial was run to establish the feasibility of coating ~1 mil matrix on thin electrodes. The 90% five micron SiC, 10% submicron SiC ink formulation was used with the existing IFC production coater. Coating thicknesses of 1.2 and 1.3 mils were obtained by increasing belt speed to the maximum and reducing curtain thickness to a minimum possible with this piece of equipment. The thin electrodes required a carrier to eliminate slipping on the acceleration table belt. Electrodes were taped to the carrier with two sided tape on the leading edge to keep them from moving under the curtain or during deceleration. The bubble pressure of a sample cut from the 4-ft^2 electrode curtain coated with 1.2 mil matrix is shown in the following table.

Table 3.3-3. Curtain Coated Thin Matrix Bubble Pressure

Bubble Pressure	Compressive Load
22 psid	50 psi
18 psid	40 psi
16 psid	30 psi
10 psid	20 psi
6 psid	10 psi
5 psid	5 psi

In the April report the importance of matrix thickness was shown for the 90% five micron SiC, 10% submicron SiC ink formulation. The bubble pressure of wire wound rod coated subscale samples varied from 8-12 psid at 5 psi axial load to 12-18 psid at 50 psi axial load. Figure 3.3-1 shows how submicron SiC matrix bubble pressure varies with thickness.

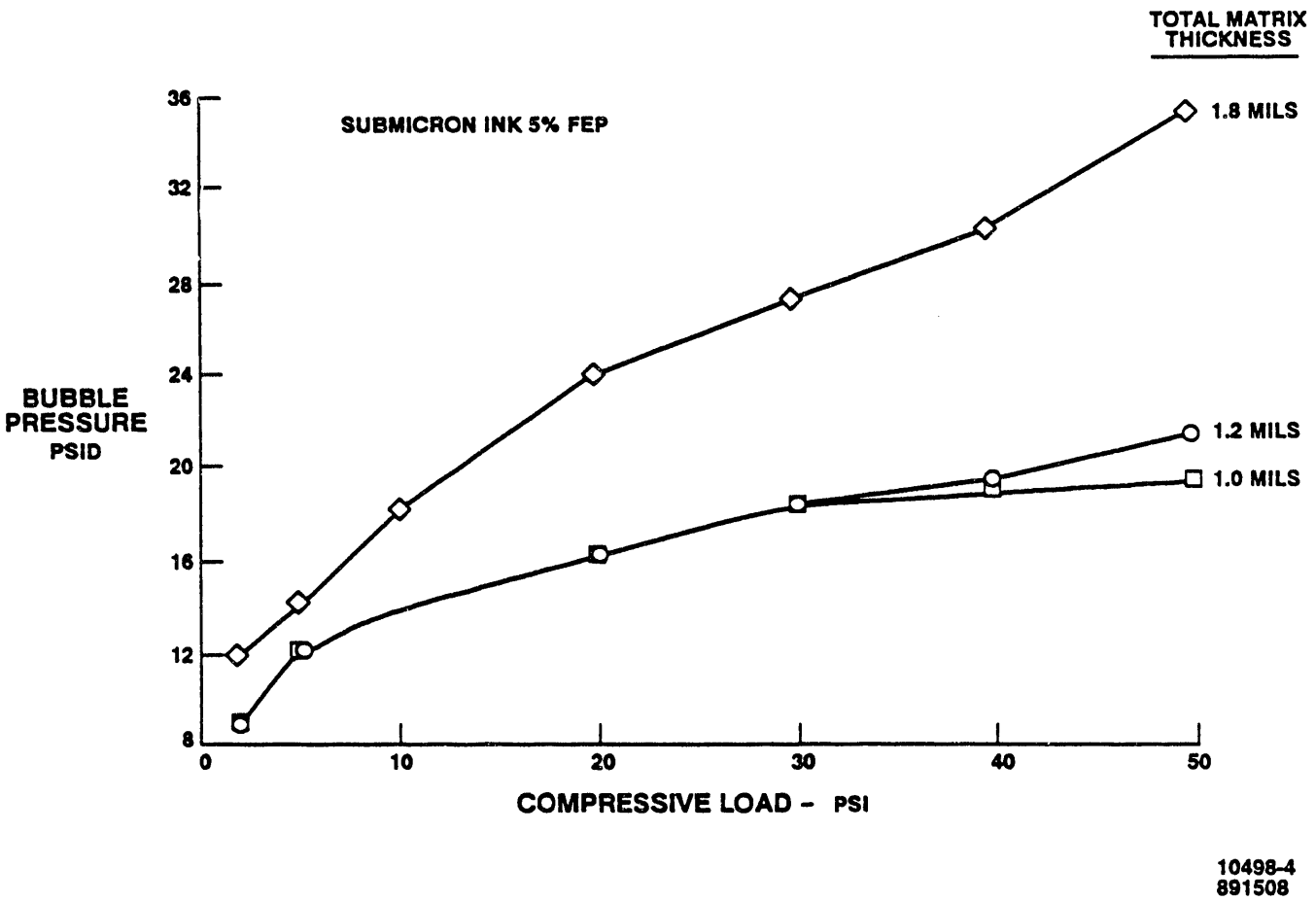

Figure 3.3-1. Thin Matrix Bubble Pressures

Subtask 3.4 - Molded Cooler

Objectives

The objective of this subtask is to develop material and process technology to fabricate molded coolers 10-ft2 in size that meet the technical requirements and cost objectives established in the conceptual design.

Activity

Subscale graphite and resin composites molded by a one-step process were post-baked to select the optimum graphite type and resin content for additional development. The composites were molded using two types of high conductivity graphites and a range of resin contents. Two samples of each composition were post-baked in separate, but identical cycles. Following post-bake, the samples were visually examined for cracks and blisters. The thermal conductivity of all samples surviving post-bake without cracks and blisters were measured. The results of these tests are shown in Table 3.4-1. The composition of sample AE1 was selected for additional development.

Table 3.4-1. Post Bake Results for Subscale Graphite-Resin Composites

Graphite Type		Nominalized Resin Content	Post Baked Cycle I	Post Baked Cycle II	Thermal Conductivity Btu/Hr- °F-Ft^2
Graphite A	AE1	1.00	Y	Y	8.6
Graphite A	AE2	1.12	Y	Y	8.1
Graphite A	AE3	1.27	N	Y	
Graphite A	AE4	1.38	N	Y	
Graphite A	AE5	1.38	N	N	
Graphite A	AE6	1.54	N	N	
Graphite B	BE4	0.85	Y	Y	8.9
Graphite B	BE3	1.00	N	Y	
Graphite B	BE2	1.15	Y	Y	6.0
Graphite B	BE1	1.38	N	N	

Legend:

Y - Survived Post Baking
N - Cracked During Post Baking

Subscale shimming trials were performed on centering the cooler tube array in the cooler composite. The shimming trials tested and evaluated several shimming techniques: straight shims of metal wire, teflon, solid graphite, phenolic-graphite and weaving metal wire between the tubes. The woven metal wire technique worked best.

Several subscale 6 inch by 6 inch coolers, molded with tube arrays, were post-baked at 630°F in a nitrogen environment for two days. No cracking or blistering of the coolers was introduced by the post-baking. The thermal stability of the post-baked parts was verified by thermal cycling a post baked cooler ten times at 400°F.

Thermal resistance measurements of subscale coolers indicated a value 2.3 times the baseline cooler. Photomicrographic examination revealed density variations throughout the thickness of the sample and a poor tube to composite bond. The sample, the forming process and the post-baking cycle are being examined to determine the cause of this poor bond.

Scale-up trials were conducted for the 31.75" x 31.75" planform size. Five samples were successfully molded; two did not contain tube arrays; one contained a standard tube array; and two contained tube arrays which had the tube exterior specially prepared to enhance the bonding of the composite to the tube. These five parts will be post-baked and examined.

Commercial graphite is being evaluated as an alternative to the baseline molded cooler holder. Three different graphite grades were evaluated in subscale 6 inch x 6 inch laminating trials to evaluate handleability, iR, and acid protection. The results are shown in Table 3.4-2. Graphite Y was selected for additional evaluation.

Table 3.4-2. Commercial Graphite Cooler Holder

Physical Properties			
Parameter	**Type X**	**Type Y**	**Type Z**
iR	**1 mV/100 ASF**	**1 mV/100 ASF**	**5-10 mV/100 ASF**
Electrolyte Barrier	Baseline Barrier	Baseline Barrier	Too dense to employ Baseline Barrier
Handleability	To weak to be machined at full size	Acceptable	Acceptable

Subscale 6 inch x 6 inch cooler assemblies were fabricated with the commercial graphite plate and a baseline holder. The arrays were formed from 7/16th inch diameter x 35 mil wall tubing. Grooves were machined to specification and inspected for compliance. Subassemblies and the final assemblies were iR tested to requirements and passed.

Testing was conducted in the 6 inch x 6 inch thermal resistance rig at identical conditions. Both test pieces had acceptable and essentially identical thermal resistance (Table 3.4-3).

Table 3.4-3

Control Holder Material	Thermal Resistance Hr Ft^2 °F/Btu
Baseline	0.0074
Commercial Graphite	0.0072

A 4-ft^2 cooler assembly was fabricated using commercial graphite plates for the holders. The array was formed from 7/16" OD x 35 mil wall tubing with a uniform pitch. This cooler was evaluated in a full-scale heat transfer rig.

The rig accumulated 18 thermal cycles and was loaded from 10 to 50 psi axial loads, which covers the axial load range of end to beginning-of-life. The test results are summarized below (Table 3.4-4).

Table 3.4-4

Axial Load PSI	Thermal Resistance Hr Ft^2 °F/Btu
50	0.0062
20	0.0067
10	0.0072

A small-area cooler incorporating a commercial graphite cooler holder was inspected after heat transfer tests. The test which consisted of eighteen thermal cycles at 350°F did not cause any cracks or degradation to the cooler.

Lamination trials of the commercial graphite samples resulted in inconsistent electrical resistance (iR) measurements. The inconsistency was traced to the variability of the open pore volumes of the samples. This data is shown in Figure 3.4-1.

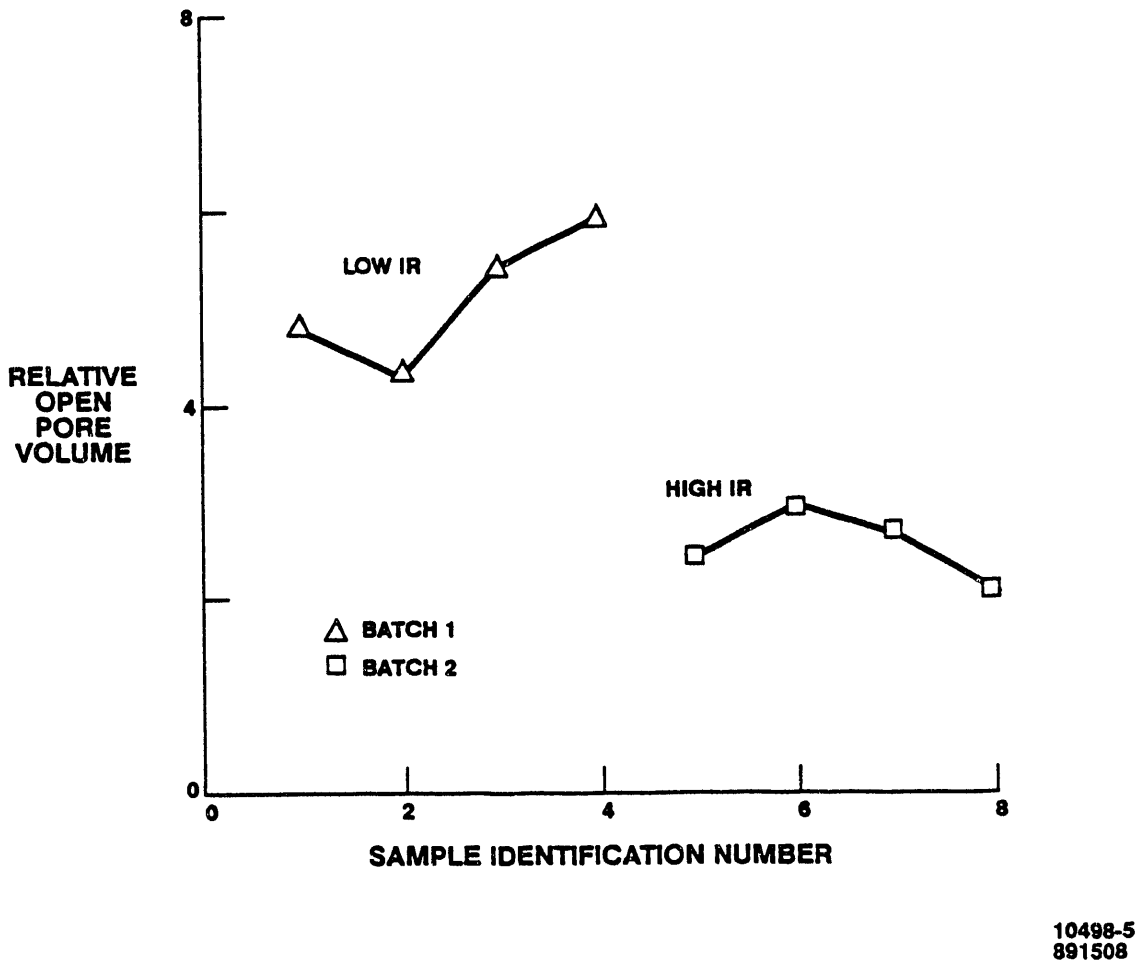

Figure 3.4-1. Relative Open Pore Volume of Commercial Graphite Samples

TASK 4 - STACK DESIGN

Objectives

The objective of this task is to update the full-scale conceptual design and to define designs for one small area development stack and three 10-ft2 subscale stacks.

Activity

Design of a prototype cooler array for the short stack rig was completed using preliminary analysis begun during the last reporting period. Initial tubing diameter for this array is 5/16 diameter.

Design of a non-molded holder for this array was completed. This holder will be machined from commercially-available graphite.

Layout drawings of the short stack rig in a pressure vessel were completed.

TASK 5 - FABRICATION

Subtask 5.1 - Repeat Components

Objectives

The objective of this effort is to fabricate the repeat components for one small area development stack and three 10-ft^2 subscale stacks each consisting of approximately 30 cells. These stacks will be of the Configuration B cell design. The repeat components consist of the substrates, electrodes, electrolyte reservoir plates, and cooler assemblies.

Activity

No activity scheduled during this report period.

Subtask 5.2 – Non-Repeat Components

Objectives

The objective of this effort is to fabricate the non-repeat components for the small area development stack and three 10-ft^2 subscale stacks of approximately 30 cells each. These stacks will be of the Configuration B cell design. The non-repeat components consist of reactant manifolds, coolant manifolds, axial load system, power take-off hardware and pressure containment vessel.

Activity

Two new types of cooler connecting hoses are being evaluated to reduce hose costs and to qualify an additional supplier. The test configuration is shown in Figure 5.2-1.

One new hose assembly type being tested is fabricated with a modified lower cost dielectric hose formulation. All other hose materials and the connecting system are identical to the baseline assembly. The second type of hose assembly employs an alternative dielectric concept and connectors. Four hoses of each type are being tested.

Test conditions are as follows:

Coolant Flow Rate	150	pph
Coolant Inlet Temperature	350	°F
Coolant Inlet Pressure	145	psig
Coolant Inlet Subcooling	13	°F
Applied Voltage	200	volts D.C.
Water Quality		
pH	6 – 8.5	
O_2 Conc.	30 – 500 ppb	
Turbidity	0.5 – 1.0 NTU	
Conductivity	$< 0.5 \times 10^{-6}$ mho's	

Presently, the hoses have accumulated 800 hours of testing.

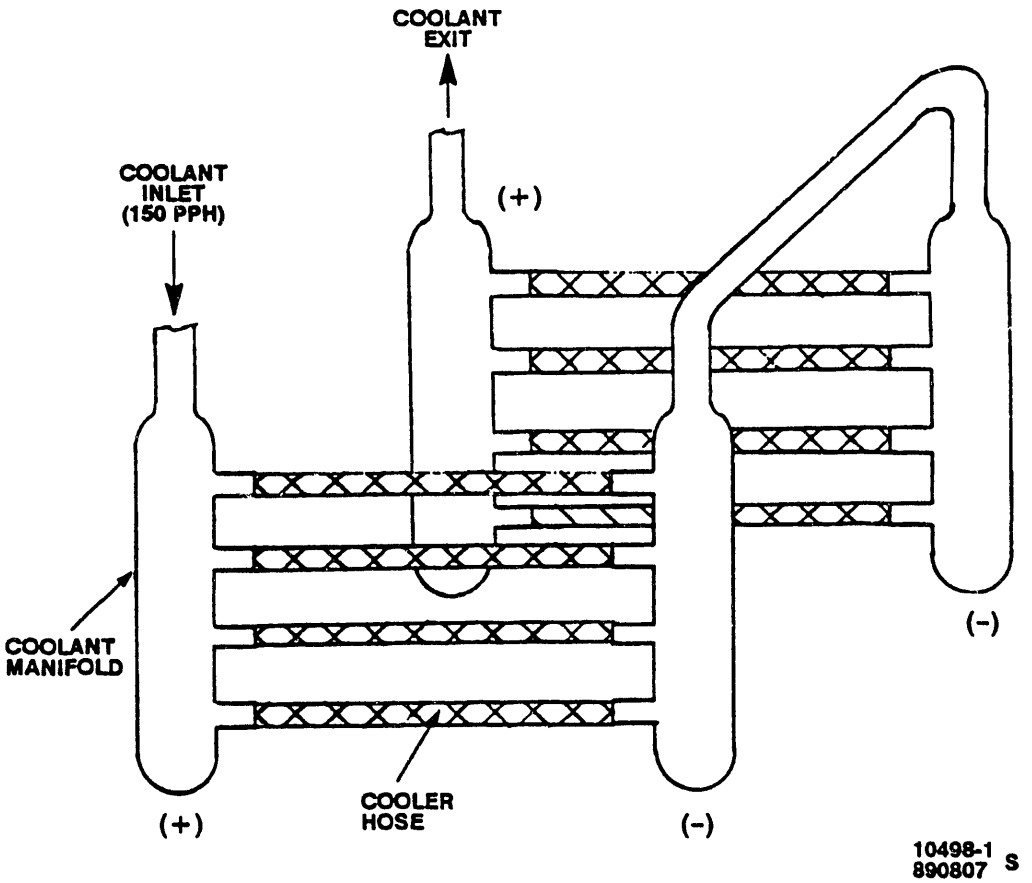

Figure 5.2-1. Cooler Connecting Hose Evaluation Set-Up

Ceramic coatings are being evaluated as a lower cost alternative to the baseline PFA coating systems. A commercial coater has provided samples with a coating judged by this vendor to be acid resistant. The coatings have been exposed to 400°F phosphoric acid at IFC to evaluate the corrosion resistance. The coatings corroded at approximately 60 mils/yr. This corrosion rate is unacceptably high. Testing at less severe conditions, 300°F in phosphoric acid, has commenced. This temperature coincides with the operating conditions in the inlet manifolds and exit piping.

PPS and Bismaleimide (BMI) are being evaluated as candidate resins for non-repeat component applications. The PPS system being evaluated is from the same supplier as the material evaluated as a coating replacement. The PPS samples tested to date contain no fillers. The BMI was formulated with an inert filler.

The results of the initial screening tests are shown in Table 5.2-1.

Table 5.2-1. Non-Repeat Resins

		Load Deflection at 1000 PSI % Thickness/Lb.		Electrical Resistance at 500 Volts/5000 PSI Ohms	
Resin	**Filler**	**Ambient**	**400°F**	**Ambient**	**400°F**
PPS	None	——	——	$> 1 \times 10^{-8}$	$> 1 \times 10^{-8}$
BMI	Yes	$< 5 \times 10^{-4}$	$< 5 \times 10^{-4}$	$> 1 \times 10^{-8}$	$> 1 \times 10^{-7}$

Subtask 5.3 – Stack Assembly

Objectives

The objective of this effort is to assemble the small area development stack and two 10–ft^2 subscale stacks of Configuration B cells for testing at 120 psia. The third 10–ft^2 subscale stack will be assembled and tested under an anticipated EPRI program.

Activity

No activity scheduled during this report period.

TASK 7 - MANAGEMENT AND DOCUMENTATION

Objectives

The objective in this task is to provide the program management necessary to maintain technical and budget control of the program activities, and to provide adequate documentation of its progress per contract requirements.

Activity

The following reports were submitted during the quarter:

- Technical Progress Report No. 8 Annual 1988 (FCR-10093) was issued on May 3, 1989.
- Monthly Technical Progress Report No. 9 for January (FCR-10171) was issued on May 3, 1989.
- Monthly Technical Progress Report No. 10 for February (FCR-10227) was issued on June 2, 1989.
- Quarterly Technical Progress Report No. 11 for January-March (FCR-10308) was issued on June 8, 1989.
- Monthly Technical Progress Report No. 12 for April (FCR-10374) was issued on June 28, 1989.
- Cost Management Report No. 11 for March (FCR-10307) was issued on April 26, 1989.
- Cost Management Report No. 12 for April (FCR-10373) was issued on May 19, 1989.
- Cost Management Report No. 13 for May (FCR-10421) was issued on June 21, 1989.

END

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