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MASTER

DEVELOPMENT OF
MOLTEN-CARBONATE FUEL-CELL TECHNOLOGY

Technical Status Report For The Period
August-October 1980

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EXECUTIVE SUMMARY

Efforts in the development of mechanically stable anodes with more enduring pore structures are being concentrated at present on our powder/metal alloy fabrication process. One of the new alloy compositions being prepared for electrode production concerns a new 50 at% nickel-copper powder. Other new compositions have been produced and, as a first stage, are being tested under decay-accelerating, high temperature conditions. Laboratory cell tests on several different high surface area anode materials are also reported which show considerably improved initial performance levels. And a bench-scale cell using a conventional Ni/Co anode has been operated for 3500 hours with completely stable performance.

Another task in the electrode development area is directed at producing low resistance cathodes. The newly developed fabrication procedure of wet-forming thin nickel powder films from aqueous PVA-binder slurries is showing promising results. Thin-cast films in the absence of reinforcing screens can have less than 5 mil thickness after H_2 -sintering at $900^\circ C$ but still maintain the strength and appearance of fairly strong gray paper. To reduce resistance in another way, the accurate volume-specific resistances of variously lithiated nickel oxide crystal structures have been measured at $650^\circ C$. The values show that catalyst layer resistivity at the cathode can be diminished from 0.015 to $0.005 \Omega \cdot cm$ if the doped lithium content can be increased threefold. However the means of maintain (in-cell) equilibrium lithium contents of 15 at% or more are not yet clear.

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The relatively low-cost, standard electrolyte of 50Li/50K cation ratio used at ERC is being re-examined for performance level compared to higher lithium content melts. All other cell components and the tile matrix material used in these tests are of exactly the same type so as to be able to positively identify the electrolyte composition effect. The fact that different blends have different melting points and melting ranges which can strongly affect tile thermomechanical behavior is being taken into account by evaluation of the rheological behavior of the structure in a simple but very effective slump test apparatus.

A second pressurized, bench-scale (300 cm²), cell test has been operated successfully showing that under the demanding conditions of 80% utilization with low-Btu fuel, the performance goal of 850 mV at 160 mA/cm² can be met with 10 atmosphere pressurization.

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TASK 1 ANODE DEVELOPMENT

The anode development effort this quarter focused on preparation and testing of additional candidate materials and testing of improved low-cost anodes in laboratory-scale and bench-scale cells.

Preparation of Anode Powders

Using our small batch (2 kg) coprecipitation method, further quantities of Ni-based hydroxides have been prepared to investigate such parameters as different spray-drying temperatures (to produce powders with greater or lesser moisture content and particle size), lower cobalt content (5 instead of 10 at%), and the addition of a major quantity of copper (50 at%) instead of cobalt. Reduction of the powders will be accomplished in our large, H₂ retort using the identical temperature/time and gas flow conditions that have previously provided good powder structures. In particular, three hydroxide batches, identical in composition, but with different spray-drying temperatures will be reduced in the same run. Thus the only variable will be the initial moisture content of the hydroxide (manifesting itself in a more agglomerated structure) which is to be evaluated for its effect on surface area and particle size after reduction to powder metal.

Accelerated Anode Stability

Initial changes in characteristics observed with four different anode structures, aged for 300 hours at 700°C in the presence of LiKCO₃ (as shown in Table 1.1), have been minor. The porosity, surface area, weight and thickness changes at 1500 hours are showing continuing small changes

TABLE 1.1 ACCELERATED ANODE STABILITY IN MOLTEN CARBONATE ENVIRONMENT

(Carbonate Li/K = 1:1 and Weight = 0.4g, Fuel Gas Ratio = 7H₂/2CO₂/1H₂O, Temp. 700°C)

TEST NO.	NOMINAL COMPOSITION	Hg-POROSITY, %			MEAN PORE SIZE, μm			BET SA, m^2/g			WEIGHT, g		THICKNESS, mil	
		pre/300/1500			pre/300/1500			pre/300/1500			pre/1500		pre/1500	
1	Ni (ERC)	64	62	63	8.4	8.8	9.2	1.3	0.6	0.5	1.8	1.8	27	30
	Ni/10Co	70	69	69	8.8	†	9.4	0.8	0.8	0.6	1.4	1.3	26	26
	Ni/10Co/5Cr	76*	70	77	5.5	4.4	3.0	0.6	1.2	3.0	1.3	1.3	26	29
	Ni/3Al	70	63	68	8.0	8.8	8.9	4.2	3.4	2.6	1.6	1.4	33	37
2	Ni(UC-287)	60	66		12	12		3.0	1.5		pre/300		pre/300	
	Ni/10Cr(ANL)	65	67		5.5	4.6		0.2	2.3		1.2	1.2	29	25
	Ni(pre 7-38)		58			4.2			3.4		1.2	1.3	30	32
	Ni/8Co/5Mo	74	70		6.7	9.5		2.4	1.6		1.0	0.9	28	26
											1.2	1.2	27	26

* Calculated geometric porosity.

† Inconsistent results; will be reanalyzed.

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in a consistent pattern. Depending on the variability of consecutive samples being taken from experimental plaques and the precision of the porosity measurement, the Hg-porosities appear relatively unaffected. However Ni, Ni/Co and Ni/Al pore dimensions increase somewhat, while Ni/Co/Cr is small initially and decreases to about 1/2 of the former pore sizes. BET-surface areas show consistent correlation with this behavior in that Ni, Ni/Co and Ni/Al (even though the latter starts off very high) all decrease while Ni/Co/Cr increases to about 3 m²/g. The variations in weight and thickness are not considered very significant because of:

- a) the variability in plaque thickness measurement, and
- b) the fact that the samples are not under compression during out-of-cell testing. As more analytical results and experience are gained, the interpretation of accelerated testing will become more meaningful; a second test with four more experimental plaques has just been initiated.

Sulfur Contaminant Analysis

As reported earlier*, a good deal of the carbonate present in the pores of an anode operated on H₂S-contaminated fuel, such as in Cell C-1 (750 hours), can become converted to a type of sulfur species which is water soluble. It was assumed this might be LiKS or LiKSO₄ but some initially anomalous results were obtained. Soluble sulfur in a piece of the post-test anode close to the exit appeared too high when compared to the sulfur content obtained on a similar anode exit specimen which was analyzed in its entirety without being washed. High sulfate content of the deionized wash water was suspected as the cause but two additional analyses with very pure water suggest that various regions of the same anode (one taken at the center and one at the

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* ERC Tech. Status Report, CHI/11304-16, Feb. - Apr. 1980.

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entry) show very different sulfur contents. Apparently a sharp gradient of sulfur concentration is established in the anode. The post-test washing procedure typically removes about 1 to 2% of soluble material (carbonates, etc.) from the anode but in this case greatly varying sulfur contents were found in the different filtrates from the various electrode areas as shown in Table 1.2. Concentration of soluble sulfur in the as-is, post-test anode varies twenty-fold from 1.8% at the entry to 0.09% at the exit while the concentration of insoluble sulfur (e.g., Ni/Co sulfides as surface species) measured in the washed specimen varies in about equal proportion but at a ten-fold lower level. If the soluble sulfur is calculated as LiKSO_4 , from 32 to 64% of the carbonate (and probably even higher at the entry) are seen to convert to a sulfur containing salt.

TABLE 1.2 ANALYSIS OF WATER-SOLUBLE SULFUR IN THE C-1 ANODE

ANODE SPECIMEN		SULFUR IN SPECIMEN		H ₂ O-SOLUBLE MATERIAL (CO_3^{2-} AND SO_4^{2-} SPECIES)		
Site	Wt, mg	% Soluble	% Insoluble	TOTAL, mg	S-species in this material	
					mg	% as LiKSO_4
Entry	222	1.8	0.16	*	4.0	*
Center	459	0.28	0.034	9.0	1.3	64
Exit	633	0.09	†	7.9	0.57	32

* Results questionable.

† Not obtained in first set of analyses.

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These data do not contradict earlier information regarding the total sulfur content in the tile (0.11%) and at the tile/anode interface (0.12%). Apparently, soluble S-species slowly exchange between the electrolyte in the anode and that in the tile, causing a more uniform sulfur concentration in the tile as this contains from 30 to 40 times as much carbonate. Once dissolved in the electrolyte of the tile, it is then very difficult to release the sulfur compounds even when operating on clean gas because of the much lower activity of the contaminant in the large volume of tile electrolyte. It seems reasonable to suggest therefore that the anodic (half cell) reaction involving H_2S :

is a very important one because of the high solubility of the $\text{SO}_4^{=}$ ion in molten carbonate and the large sink of carbonate that backs up the anode. Thus much of the H_2S may be scavenged from the entering gases early in the anode chamber forcing the $\text{H}_2\text{S}/\text{H}_2$ ratio to remain low in the thin gas layer flowing toward the exit and thus causing less sulfate absorption toward the outlet. No further sulfur testing is planned in the near future.

Laboratory-Scale Cell Testing

One previously started cell and three new cells were operated this quarter with different anode materials (see Table 1.3).

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TABLE 1.3 LABORATORY-SIZE CELL TESTING

CELL	ANODE	BET SA, m ² /g	PEAK PERFORMANCE @ 115 mA/cm ² , mV
149	Ni/10Co	0.24	865
151	Ni/10Co	0.24	825
152	Ni/3Y	0.7	850
153	Ni/10Co	0.5	880

Performance continues to improve because of anode improvements as discussed below.

Cell 149: This cell was assembled last quarter with a HSAG matrix (DLB-1)*, a standard SS-screen reinforced Ni-cathode and an optimized Ni/10Co anode. Its initial performance was better than any of our earlier state-of-the-art cells but it showed a gradual performance loss accompanied by increased resistance, as illustrated in Figure 1.1. During the first 200 hours of testing, the cell resistance increased from less than 0.4 to 0.53 $\Omega \cdot \text{cm}^2$. It was then stable from 200 to 550 hours after which it gradually increased to 0.64 $\Omega \cdot \text{cm}^2$. The first increase is thought to be due to initial equilibration and settling of components but the second gradual increase may have been due to continued anode sintering causing contact problems between the electrode and tile; post-test anode thickness could only be estimated by difference since the anode and current collector were tightly fused together. Further diagnostic tests and complete analytical characterization are still in progress and will be discussed later.

* For further details of cell characteristics refer to Table A in the Appendix.

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Std. Conditions at 650°C
 Anode Gas: 74 H₂/19 CO₂/7 H₂O
 Cathode Gas: 15 O₂/30 CO₂/55 N₂
 Gas Flows: 3A equivalent

FIGURE 1.1 LIFEGRAPHS OF CELLS 149 and 151

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Cell 151: This cell was also assembled with standard components although the Ni/10Co anode was not prefilled with electrolyte. A different tile very similar to the previous (Cell 149) HSAG-matrix was used (DLB-1,2)*. The cathode consisted of an identical, SS-screen-reinforced, Ni electrode with a preoxidation porosity of 75% and mean pore size of 10.5 μm . The cell displayed good OCV (1.08V), and wet seal behavior†. Its initial performance (825 mV at 115 mA/cm²) was not as high as that of Cell 149. But, as shown in Figure 1.1, performance of Cell 151 finally stabilized at a slightly higher level than for Cell 149. This cell was also terminated at ~1500 hours; further analysis of the components is still in progress.

Cell 152: This cell was assembled to test a nickel anode containing 3 at% coprecipitated refractory metal (Plaque PR-6B)*. The HSAG tile (Batch DLB-1,2)* and stainless steel screen reinforced Ni cathode (Plaque 27R-5)† were similar to those of Cell 151. The new Ni/3Y anode cell displayed good OCV (1086 mV), good wet seal efficiencies and good performance.† The performance gradually decayed but then stabilized around 1000 hours at 780 mV (115 mA/cm² under standard conditions) at which time the test was terminated. Post-test analyses and diagnostics with particular emphasis on anode structural changes and carbonate content are not yet complete.

Cell 153: This cell was assembled with a new, higher surface area (0.5 m²/g) Ni/Co anode* which also contained prestored carbonates. The tile utilizes a HSAG lithium aluminate matrix (DLB-1,2) with the same characteristics shown above and the cathode is a standard, 20 mil thick,

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* Refer to Table A in the Appendix.

† Refer to Table B in the Appendix.

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SS-screen reinforced electrode pressed to 74% preoxidized porosity. Thus Cell 153 is constructed with components very similar to those of Cell 149. It differs only in the higher surface area of the somewhat thinner anode. This cell displayed even better initial performance than Cell 149: about 15 mV more at 115 mA/cm² under standard gas and flow conditions.

Bench-Scale Cell Testing

Two bench-scale cells were operated for the primary purpose of endurance testing of anodes; these are Cell 7-36 with the second large, bench-scale, Ni/10Co anode and Cell 7-38 with a specially treated Ni anode.

Cell 7-36: The large Ni/10Co anode (~300 cm² surface area) is being tested in association with proven cathode, tile and hardware components and continues with good endurance. It has accumulated ~3500 hrs of stable performance. Figure 1.2 shows that although not exceptionally high, cell potential under standard gas and flow conditions has stabilized at 725 mV. Cell resistivity in the first 2000 hours of life decreased to about one half (0.7 $\Omega \cdot \text{cm}^2$) of its initial value but lately appears to be stabilizing at 1.0 $\Omega \cdot \text{cm}^2$.

Cell 7-38: This performance reference cell which again uses standard cathode, tile and hardware components has also been continued. Initial performance was compared in a previous report* with an earlier state-of-the-art cell. Constant conversion data have now been taken at four sets of anode (A) and cathode (C) gas utilizations:

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* CHI/11304-17, May-July 1980, p. 19.

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FIGURE 1.2 LIFEGRAPH OF CELL 7-36

M0099R

75A/50C; 50A/50C; 75A/25C; and 50A/25C. Figure 1.3 illustrates cell performance in terms of potential vs current density at constant 75% and constant 50% fuel conversions. Since oxidant conversion showed much less effect on performance than fuel conversion in this particular cell using a standard SS-reinforced cathode, the points on the curves are shown as short line segments representing average values for the range of 25 to 50% oxidant conversion. Overall, there is an approximately constant loss (i.e., a delta between the oxidant-averaged, fuel utilization curves) of ~40 mV associated with the 25% increase in fuel utilization. Further development may have to be focused on improving gas permeation and exchange in the anode pores.

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M00126

FIGURE 1.3 CONSTANT FUEL CONVERSION PERFORMANCE IN CELL 7-38

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TASK 2. CATHODE DEVELOPMENT

Cathode Fabrication

During this quarter, work on the development of cathode structures was further directed toward the refinement of our dry and wet forming techniques. Reliable production procedures yielding consistently good quality electrodes were successfully established. Electrode lots with different specifications were also produced. The initial characterizations for these lots are included in Table 2.1. Uniformity in strength, thickness, porosity and dimensions of the pieces included in each lot was good and no obvious cracking was observed.

The successful, screen/film sandwich, fabrication procedure that has yielded these uniform results consists of casting nickel films from an aqueous slurry containing a polyvinyl alcohol binder. Either one or two such films

TABLE 2.1 CATHODE PRODUCTION PROCESS DEVELOPMENT

LOT	FORMING METHOD*	NO. OF PIECES (8x8")	REINFORCEMENT MESH WIRE SIZE, in.	THICKNESS, mil	GEOMETRIC POROSITY (SCREEN CORRECTED), %
1	↑ DRY ↓	4	20x20x.005	12	78 (81)
2		4	20x20x.005	14	78 (80)
3	↑ WET ↓	3	20x20x.005	7	78 (84)
4		14	60x60x.0045	15	72 (81)
5		5	none	4.5	78
6		5	↓	6	79
7		5		13	75
8		5		15	73
9		5		18	77
10		14		24	72 (77)

* Post-formation treatment in all cases includes H₂-sintering at 900°C for 40 minutes.

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are then sandwiched in a partly dried, tacky condition onto the SS-reinforcement screen to form the "green" electrode structure. Subsequently the electrode is further air-dried and ultimately sintered in a hydrogen atmosphere at 900°C for 40 minutes.

NiO Lithiation

Lithiated NiO powders were prepared by our subcontractor, Westinghouse Electric Corporation, with Li-contents ranging from 1 to 15 atom%. The powders were prepared by solution reaction of the required amounts of nickel carbonate and lithium carbonate with formic acid. Solutions were then evaporated by radiant heating and freeze-drying of the product. The bright green powders thus obtained were decomposed at 300°C under pure O₂ atmosphere to produce black, lithiated, nickel oxide powders. A schematic of the preparation procedure is shown in Figure 2.1.

110131 a

FIGURE 2.1 LITHIATED NiO POWDER PREPARATION
(Formate Process)

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X-ray measurements and wet analysis results (see Table 2.2) indicate that with this low temperature formate decomposition process all the available lithium enters the oxide crystal lattice.

TABLE 2.2 LITHIUM CONTENT IN VARIOUS PREPARATIONS

SAMPLES	Li CONTENT, at%	
	By X-ray Lattice Parameter	By Wet Analysis
Cell 7-31 (avg. of 2 post-test samples)	3-3.5	1.8*
5% Li preparation	4-5	4.6
15% Li preparation	14-15	14.7

* Low analysis due to inadequate exclusion of screen corrosion products from used electrode material.

Electrical Conductivity

Electrical conductivity samples were made from the lithiated nickel oxide powders prepared as described above. They are pressed into a pellet 1/2 in. OD x 1/2 in. long in a single action, split die at ~345 MPa (50,000 psi) pressure. Sintering at 1000°C for 36 hours in an oxygen atmosphere produces compacts of 93% theoretical density.

A 2.2 mm slice is cut from the center of the pellet and a 2.2 mm center section is cut from this slice. The sample is hand-lapped on 600 grit paper to yield a bar 2 mm on its sides and approximately 1/2 inch long. Sample preparation is finally completed by vapor depositing rings of Pt for contacts on each end and at 1/3 the length of the bar (Figure 2.2).

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FIGURE 2.2 ELECTRICAL CONDUCTIVITY TEST SAMPLE

Volume specific resistances were obtained by Westinghouse under 100% O₂-atmosphere in the ceramic enclosure of a tube furnace held at a number of controlled temperatures. Figure 2.3 presents the data showing that below 250°C, the doped nickel oxide conducts in an anomalous fashion with higher room temperature resistance for the 15 at%

FIGURE 2.3 VOLUME-SPECIFIC RESISTIVITY OF LITHIATED NiO COMPACTS

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lithiated powder compared to the 5 at% lithiated material. Apparently some kind of phase or structure transformation occurs between 150° and 250°C which inversely affects the resistivity changes due to doping below and above that temperature range. In reviewing the literature it was indeed found, as Van Houten* reports, that at room temperature NiO is slightly rhombohedral and at the Néel point (250°C) becomes completely cubic. It can therefore be concluded that more consistent results are obtained when resistances are measured near the MCFC operating temperature. The data show that 15 at% lithium doping lowers nickel oxide-resistance approximately threefold compared to the 5 at% level. The latter is close to the level normally found in post-test cathodes. In-cell tests are needed to determine if the higher Li-levels can remain stable over the life of a cell.

Prelithiated, Preoxidized Cathodes

Initial attempts to prepare cathodes from lithiated NiO powder were successful after methods were developed to increase the "green" strength of the dry packed, pressed and sintered plaques, thus providing more easily handled plaques. Adherence of the powder particles to the screen and to each other was improved in two ways: First the screen was stiffened by pressure welding the wire crossings. This also has the advantage of decreasing the total thickness of the nominal 10 mil wire reinforcement. Subsequent heating of the screen in air then aids in pre-annealing the pressure welds and is also helpful for better adherence of NiO to the slightly preoxidized surface. Secondly, the adherence of oxide particles to each

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* Van Houten, S, J. Phys. Chem. Solids, 17 (1/2), 1960.

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other was improved by the use of a small amount of Carbowax No. 200 binder which intimately holds the particles together before the plaque is final-sintered at 800°C for 12 hours in pure oxygen atmosphere.

Plaques formed in this manner are solid, adherent structures but are somewhat brittle and become slightly warped on firing. A typical plaque had a total porosity of 62 vol% with $\sim 1 \mu\text{m}$ mean pore size and a BET surface area of $0.77 \text{ m}^2/\text{g}$. Although most of these characteristics are adequate, further work is contemplated to greatly increase the MPS which is still much too small.

Laboratory-Scale Cell Testing

In-cell testing of a thin cathode structure* was begun this quarter in Cell 150. The OCV and wet seal on the anode and cathode sides were good, but cell performance was low with a higher resistance than expected ($0.68 \Omega \cdot \text{cm}^2$). The poor performance at present is attributed to electrode flooding. In connection with the $3 \mu\text{m}$ MPS of the Ni/10Co anode used, the original metallic MPS of the thin cathode appears to have been too small. The test was terminated at 590 hours and post-test observations were as follows:

- The cathode showed a thickness increase of 1 to 2 mil, similar to standard thickness cathodes. Therefore no change in cathode protrusion during assembly is needed. Also lack of contact was not the reason for the high cell resistance.
- The cathode was completely oxidized yet mechanically strong, as expected.

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* Refer to Tables A and B in the Appendix for characteristics and performance.

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- However the cathode current collector was heavily corroded, its perforations filled with rust, clearly because of the presence of excessive electrolyte. This corrosion would account for the high cell resistance. Diagnostic analyses are in progress to determine what cathode structural changes took place.

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TASK 3 ELECTROLYTE DEVELOPMENT

Study of Electrolyte Compositions

The standard electrolyte blend at ERC for a number of years has been a carbonate mix of 50Li/50K cation ratio. This relatively low cost, congruent-melting mixture has served its purpose well but a number of cells have now also been operated with the 62Li/38K cation blend which is the eutectic composition on the Li-rich side of the phase diagram. It was selected because lithium is the most active cation present and is capable of both combining with the SS-hardware components of the cell and doping the NiO cathode. Thus it should be present in excess to guard against lithium depletion in the electrolyte. Possible advantages of increased lithium content are performance improvements due to better ionic conductivity and/or electrode activation kinetics. But at ERC and elsewhere, tests to differentiate clearly between carbonate blends have usually been inconclusive because they often have involved variations in matrix and electrode types with consequent changes in such parameters as pore size dimensions, etc. Such changes can strongly influence cell performance and hence mask any effect due to electrolyte composition. Even when the same matrix batch is used, it has been found that care must be exercised to adjust the liquid content of the tile (i.e., carbonates) based on the thermomechanical characteristics of the electrolyte/matrix combination. As discussed in the next section, our present study takes into account such tile rheology changes in the form of tile slump behavior.

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The test plan and properties of electrolyte compositions and tile characteristics used in the present study are detailed in Table 3.1. The various compositions are being investigated in otherwise completely identical cells. The first three compositions were chosen to study the effect of increased lithium content without regard to melting point. When mixtures richer in lithium than the congruent or eutectic melting blends are used, however, the melting point range increases because the electrolyte separates into two fractions of different solidus and liquidus composition. The 68Li/32K electrolyte therefore contains some solid material at temperatures as high as 540°C, which is only ~100°C below the cell operating temperature.

TABLE 3.1 TILE CHARACTERISTICS IN ELECTROLYTE COMPOSITION STUDY

CARBONATE COMP. Li/K cation ratio	Mpt, °C (range)	LIQUID CONTENT IN DLB-1,2 TILE, wt%	PDE*, %	CELL TEST	COMMENTS
62/38	488	62	5	153	Binary eutectic, high-Li.
50/50	504.5	64	7	154	Congruent melting binary.
68/32	500 (460-540)	63	6	155	Off-eutectic binary Li-rich side.
65/30 (+5Ba)	480 (460-500)	65.5	6	156†	Off-eutectic ternary Li-rich side.

* PDE (percent diameter expansion) of 1-1/2 in. diam. tile under fixed temperature/pressure/time conditions.

† Not operating yet.

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A ternary electrolyte mix with increased lithium content is now being sought which will provide the same or lower melting point range than that of the sharp melting eutectic or equimolar mixtures. Barium was chosen as the third element because of the work of Davtyan et al* which shows it to be a stable melting point lowering additive. Figure 3.1 illustrates the effect on electrolyte melting range which 5 at% barium (calculated on cation content) has when blended with the (68Li/32K) off-eutectic mixture. The melting point behavior was investigated with a

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FIGURE 3.1 CARBONATE MELTING POINTS DETERMINED BY DIFFERENTIAL THERMAL ANALYSIS

* Davtyan, O.K., Terterin, G.A. and Uminskii, M.V., "Some Physico-Chemical Characteristics of Low-melting Carbonate Electrolytes", Sov. Electrochem. 6, p. 773-9, 1970.

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Dupont-DTA apparatus using carefully blended and pre-melted samples which were always heated and analyzed under 100% CO₂ atmosphere. It can be concluded that the addition of 5 at% Ba to the parent (off-eutectic) binary lowers the nominal melting point by 20°C and the maximum of the melting range by 40°C.

Thermomechanical Testing

As shown in Table 3.1, one ternary and three binary carbonate compositions have been slump tested in tiles containing our HSAG matrix (see Appendix Table A for DLB-1,2 characteristics). One further slump test was also done with a 75Li/25K cation ratio as illustrated in Figure 3.2 which compares PDE vs wt% carbonate in the tiles. Typically, PDE appears to increase logarithmically with increasing carbonate content except for the 68Li/32K and 75Li/25K mixtures over the range tested. Although more data are needed to unequivocally verify this, the sharp melting 62Li/38K eutectic and 50Li/50K equimolar mixtures (and probably also the intermediate behaving ternary) appear to give rise to a much more pronounced effect on tile slump behavior over a narrow range of carbonate content than the wider melting (off-eutectic) blends. Thus a strong influence on tile rheology is suggested by the fluid changes in the liquid phase held interstitially between the aluminate, load-bearing, particles of the matrix. Apparently these melting point effects are still felt at 650°C, a temperature well above the true melting point of the bulk mixtures (i.e., 488° and 505°C respectively for the eutectic and congruent melting phases). Thus, even though it is a simple apparatus, the ERC slump test procedure continues to prove itself a sensitive tool for testing sharp changes in MCFC tile rheology.

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FIGURE 3.2 SLUMP TEST RESULTS WITH DLB-1,2 MATRIX MATERIAL

The mechanical behavior of a tile is dependent on and can be varied by adjusting both the quantity and composition of the carbonate content. Our present experience with HSAG matrix material, has indicated that optimum cell behavior is attained when providing from 5 to 8% PDE (measured at the same 650°C temperature at which the cell operates). This has meant that tiles using eutectic carbonate compositions have to be more carefully compounded than non-eutectic blends to fall within the required PDE range. This correlation of PDE with cell performance has been observed in several tests. For

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instance, the tile used in Cell 138, containing 60 wt% liquid, was too dry and the one used in Cell 143, containing 65 wt% liquid, was too wet (slumping easily and causing initial electrode flooding). It was then expected that the tile used in Cell 147, containing 63 wt% liquid, would show optimum behavior but because its Li/K ratio had also been changed to 62Li/38K, it was again too soft. Consequently the cathode became badly flooded, as confirmed by post-test analysis.

Fabrication of Large HSAG Tiles

Laboratory-scale (8.5 cm²) HSAG-LiAlO₂ tiles have been successfully tested out-of-cell and have operated well in laboratory cell tests when carbonate content is carefully adjusted for correct thermomechanical behavior. This quarter, fabrication of bench-scale (300 cm²) tiles from HSAG-LiAlO₂ material (Batch DLB-1,2) was initiated.

Table 3.2 lists a number of these electrolyte tiles made by modifying hot pressing procedures. Tiles 88 thru

TABLE 3.2 BENCH-SCALE HSAG TILE FABRICATION

TILES, J-series	HOT PRESSING CONDITIONS				THEOR. DENSITY, %	VISUAL INSPECTION* (removal of graphite layer)		
	Pressure, MP	Temperature, °C	Time, min.	Cooling Details		Before	After	
88	49	470	30	Std procedure developed for in situ and high β-phase materials	98	Good	Microcracks	
89	49	470	30		99	Chipped	"	
90	49	470	30		" "	99	Chipped	"
91	49	470	30		" "	-	Cracks	-
92	49	480	30	" "	98	Good	Microcracks	
95	45	475	40	Slower Cooling	-	Some cracks in ctr.	-	
96	45†	465	60	<1°C/min cooling rate, improved insulation	96	Cracks at one edge	-	
98	45†	465	60	As for Tile 96 but cooled without pressure	96	Good	Lg. thickness variation (±6 mil)	
99	45†	465	60	As for Tile 98 but with thinner matrix	97	Good, one corner crack	Very uniform thickness	
100	45†	465	60	As for Tile 98	97	Good	Good with uniform thickness	

* Cracks, chips, flatness and structural irregularities.

† After reaching 465°C, pressure was increased in three 15-minute steps and held at max. P. for 15 min.

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92 were initially made using the standard procedure established for fabricating high β -phase structures. However after the removal of the mold release compound from the surface, these tiles showed major deficiencies (i.e., chipping and microcracks). This may have been caused by the finer, more spherical shape of γ -LiAlO₂ particles compared to the larger rod-shaped particles of β -LiAlO₂. After a few preliminary trials, five more tiles were produced (Nos. 95, 96, 98, 99 and 100) with progressively better characteristics while certain procedural changes were being made. The cooling rate was modified to reduce thermal stresses after the tile becomes set at high temperature. Also longer equilibration periods were used between stepwise increases in pressure to compensate for the somewhat lower final pressure and temperature used. Better quality insulation was also added around the tile mold to decrease any lateral temperature gradient across the tile.

Tiles 95 and 96 were uniform in thickness and of high density but showed some cracked areas. As is standard procedure when pressing high β -phase material, these tiles were cooled under slight pressure, ≈ 3 MPa (400 psi), which may have been too high to allow for free contraction between the mold surfaces. Subsequently, Tiles 98, 99 and 100 were made using only mold platen weight during the cooling period. These tiles were substantially free of microcracks. Tile 100 is highly uniform in thickness and was used in Cell 7-35.

Pressurized Cell Testing

A second pressurized cell (No. P7-2) was tested this quarter. The test facility was further improved as reported in our last quarterly report*. Cell hardware was also modified to improve cell performance in the following ways:

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* CHI/11304-17.

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- A nickel anode current collector was provided to reduce contact resistance.
- An increased number of support ribs were provided for better current collection and support of the nickel current collector.
- Increased gas chamber depth was designed in the cell to increase residence time.

The anode was a new, HSA ($0.6 \text{ m}^2/\text{g}$) Ni/10Co electrode (26 mils thick with 65% porosity). The cathode was a standard SS-reinforced electrode (15 mils thick with 80% porosity) and the matrix was HSAG-LiAlO₂. During the continuous pressurizing procedure, momentary stops were made at different pressure levels to measure cell resistance. As expected, the resistance was found to be constant.

A performance comparison of Cell P7-2 with similar component cells (153 and 7-32) is presented in Figure 3.3. Performance of Cells P7-2 and 153 is comparable,

FIGURE 3.3 IR-FREE PERFORMANCE COMPARISON IN RECENT CELLS

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indicating a successful scale up. Cell 7-32 was an earlier standard Ni/Co anode cell. Both of the later cells (P7-2 and 153) had shown improvements over this predecessor.

Performance data were taken at 1, 3, 5 and 10 atm (absolute) for Cell P7-2 with both high and low-Btu fuel (see Figures 3.4 and 3.5). The performance at 160 mA/cm² with high-Btu fuel was 764 mV at atmospheric pressure and 845 mV at 10 atm; with low-Btu fuel the performance was 748 mV at atmospheric pressure and 850 mV at 10 atm. Table 3.3 provides a summary of performance gains above atmospheric pressure at 3, 5 and 10 atm with both low and high-Btu fuels. Observed OCV gains agree with the theoretically-predicted Nernst values. Included in this table is a comparison of the

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FIGURE 3.4 EFFECT OF OPERATING PRESSURE ON THE POLARIZATION CHARACTERISTICS OF CELL P7-2

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FIGURE 3.5 PRESSURIZATION OF CELL P7-2
WITH LOW-BTU FUELTABLE 3.3 SUMMARY OF PERFORMANCE INCREASES ABOVE AMBIENT PRESSURE
FOR CELL P7-2

PRESSURE ABS. ATM.	VOLTAGE GAINS AT OCV, mV			VOLTAGE GAINS AT 160 mA/cm ²		
	THEORETICAL	LO BTU	HI BTU	LO BTU	HI BTU	
					CELL P7-2	CELL P7-1
3	22	18	21	57	25	-
5	32	31	36	83	57	83
10	46	44	44	102	81	106

* Oxidant Utilization: (Air/CO₂)
 Fuel Utilization: 80% for low-Btu and 75% for hi-Btu.

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hi-Btu gains for Cells P7-1 and P7-2. The OCV gains for both cells were substantially the same. But gains at 160 mA/cm² were somewhat greater for P7-1 than for P7-2. Apparently, the magnitude of the gain is dependent on two parameters: current density and cell design. When data from two different cells are compared at the same current density, the gains may be taken as a qualitative measure of how cell design affects performance. The less gain, the better the cell design.

Another way of evaluating improvements due to pressurization is to compare the breakdown of cell overpotentials at 1, 5 and 10 atm, as was done for the high-Btu fuel case, presented in Table 3.4. This breakdown reveals that the performance gain with pressure is due to both OCV gains and a decrease in the combined anode and cathode overpotentials. Gains due to pressurization can also be graphed vs the log of pressure and be broken down into the same components as was done for the low-Btu case in Figure 3.6. This figure shows that experimental OCV gains closely follow the theoretically calculated Nernst relationship. But total gains due to pressurization (which includes

TABLE 3.4 BREAKDOWN OF CELL P7-2 OVERPOTENTIALS
(at 160 mA/cm²)

ANODE GAS: .74 H₂/19 CO₂/7 H₂O; 75% Utilization
CATHODE GAS: 15 O₂/30 CO₂/55 N₂; 50% Utilization

PRESSURE, atm.	1	5	10
OCV, mV	1090	1126	1134
• Nernst Loss*, mV	- 115	- 115	- 115
	975	1011	1019
• A+C Polarization†, mV	- 126	- 104	- 89
IR-FREE POTENTIAL, mV	849	907	930
• IR-loss, mV	- 85	- 85	- 85
MEASURED CELL POTENTIAL, mV	764	822	845

* Estimated by ERC-W1 model considering gas activity losses due to incremental product/reactant dilution in gas chambers.

† Combined electrode polarization losses by difference.

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FIGURE 3.6 GAINS DUE TO PRESSURIZATION
 WITH LOW-BTU GAS

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reduction in overpotential due to electrode activation and diffusion mechanisms) are represented by a logarithmic pressure relationship 2.5X the Nernst slope up to somewhere between 5 and 10 atm. A limit in the pressure gains then apparently sets in shortly after 10 atm, probably due to some limiting mechanism.

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The highlights of ERC's second pressurized test using a 300 cm² bench-scale cell are:

- OCV gains are proportional to $\frac{1}{2} \cdot \frac{RT}{2F} \ln P$ as expected from theory.
- Total pressurization gains, including reductions in activation and diffusion polarization, are also logarithmic up to 10 atm and proportional to about $1.25 \times \frac{RT}{2F} \ln P$.
- Operation in a relatively large cell under the demanding conditions of low-Btu fuel and practical gas conversion has shown that the 850 mV performance goal at 160 mA/cm² current density can be achieved with 10 atm pressurization.

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TASK 4. CELL MODELING

Electrolyte Distribution Model

Improvement of the electrolyte distribution model has been completed by incorporating appropriate anode contact angles. Although further evaluation is in progress, for the time being, 33° will be used as a reasonable value for the contact angle. This electrolyte distribution model will be utilized in conjunction with the performance model described below.

Performance Model

A performance model that considers complete pore size distribution of all components and their electrolyte inventories has been formulated. The model consists of a combination of simplified thin film and flooded pore models where the contributions of activation and diffusion polarization are considered separately. A detailed description of the model follows:

The overall cell voltage of an operating cell is given by the general expression:

$$V = E - \eta \quad (4.1)$$

Where,

E is the Nernst or open circuit potential and

η is the overpotential when the cell becomes polarized under load.

The term η consists of three different types of polarizations

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$$\eta = \eta^a + \eta^r + \eta^d \quad (4.2)$$

Where,

η^a is activation polarization

η^r is ohmic polarization, and

η^d is the diffusion or concentration polarization

η^r can be expressed as

$$\eta^r = Ir \quad (4.3)$$

Where,

I is operating current and

r is internal resistance in ohms.

Activation and diffusion polarizations exist at each electrode and hence should be compared separately for each electrode. The porous structure of an electrode consists of many different size pores, and its electrolyte inventory in these pores is determined by the capillary model previously discussed. Depending on their size, some pores (smaller in diameter) may be completely filled with electrolyte and are called flooded pores while some (larger in diameter) are called "thin film" pores which may be partially filled, exhibiting a very thin electrolyte film in the portion which is empty. For each of the two cases, polarization can be expressed differently as shown below.

Performance in "Thin Film" Pores: A relationship between activation polarization (η^a) and current (I) is given by the Butler-Volmer Equation

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$$I = i_o \left[\exp \frac{\alpha_a F}{RT} \eta^a - \exp \frac{\alpha_c F}{RT} \eta^a \right] 2\pi R_p L N \quad (4.4)$$

Where,

i_o is exchange current density, A/cm² of real area

α_a is anodic transfer coefficient

α_c is cathodic transfer coefficient

R_p is pore radius, cm

L is pore length, cm

N is number of pores

The diffusion polarization (η^d) is expressed by

$$\eta^d = \frac{RT}{nF} \ln \left(1 - \frac{I}{I_L} \right) \quad (4.5)$$

Where,

I_L is the limiting current in amperes and can be obtained from the generalized pore geometry shown in Figure 4.1.

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FIGURE 4.1 GENERALIZED PORE GEOMETRY TO DETERMINE I_L

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which in terms of ionic flow through the film provides the expression

$$\frac{D_F}{\Delta} \cdot H \cdot p_A = \frac{i_L'}{nF} \quad (4.6)$$

Where,

D_F is diffusivity in the liquid film, $\frac{\text{cm}^2}{\text{sec}}$

Δ is film thickness, cm

H is Henry's constant, $\frac{\text{moles}}{\text{cm}^3 \cdot \text{atm}}$

p_A is partial pressure of limiting species A, atm.

i_L' is limiting current density, A/cm² of real area

and

$$I_L = i_L' \cdot 2\pi R_p L N \quad (4.7)$$

Performance in Flooded Pores: Activation and diffusion polarizations can be obtained in the same way as in the thin film model (Equations 4.4 and 4.5), except that the limiting current is obtained from the expression*:

$$I_L = 4\pi nF D_F H p_A N \sum_{i=1}^{\infty} \frac{\tanh \lambda_i L}{\lambda_i}$$

* Wasan, D., Schmidt, T. and Baker, B., "Mass Transfer in Fuel Cells, Part I - Models for Porous Electrodes," Chem Engr. Progress Symp. Series, 63 (No. 77), p. 16-25, 1967.

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where Eigen values λ_i are the roots of the Bessel Equation:

$$J_0(\lambda_i, R_p) = 0$$

Assumptions underlying these equations are:

- Concentrations of reactants and products at each electrode are assumed uniform and equal to the average of inlet and outlet concentrations.
- Inside a pore, gas phase mass transfer resistance is neglected.
- Film thickness is uniform.

The model will take into account complete pore structure distributions of all cell components rather than assuming a uniform "average" pore size. Pores with diameters smaller than "equilibrium pore diameter" will be assumed flooded. Pores larger than the equilibrium pore diameter will be assumed coated with a thin film. Both the flooded pore and thin film pore sections of an electrode will be further divided into subsections. Each subsection will have a representative pore diameter and pore volume contributed by its pores. For each subsection, η^a vs I and η^d vs I plots will be prepared, using the corresponding model equations. Then by summation of current contributed by each subsection, η^a vs I and η^d vs I plots can be obtained for the whole electrode. For each electrode η^a and η^d can be obtained at operating current, I , which will then give total η^a and η^d of the cell. Cell voltage can now be computed as:

$$V = E - Ir - \eta^a - \eta^d$$

A computer program will be developed to perform these operations using the above modeling equations to predict cell performance.

APPENDIX
TABLE A

SUMMARY OF CELL COMPONENT CHARACTERISTICS

CELL NO.	ELECTRODES							ELECTROLYTE							PURPOSE OF CELL TEST	
	COMPOSITION	THICKNESS, mil	ANODE		Ni CATHODE (SS-Reinforced)			BATCH NO.	MATRIX			COMPOSITION		TILE THICKNESS, mil		
			MPS, μm	CALC. POROSITY, %	THICKNESS, mil	MPS, μm	CALC. POROSITY, %		LiAlO ₂ MOL%	BET SA, m ² /g	LIQUID, Wt%	Li/K ION RATIO				
LAB - SCALE																
149	90Ni/10Co	31	3	65	21	10.5	75	DLB-1	6	0	94	12	62	62/38	75	Advanced high- γ matrix with optimum content selected from slump tests.
150	90Ni/10Co	33	3	65	11	8.3	78	DLB-1,2	4	0	96	13	63	62/38	75	Thin cathode (11 mil) test.
151	90Ni/10Co	30	3	65	21	10.5	75	DLB-1,2	4	0	96	13	62	62/38	76	Reference cell for DLB-1,2 Batch tiles.
152	Ni/3Y	32	8	67	29	11	78	DLB-1,2	4	0	96	13	62	62/38	77	Nickel anode with added refractory oxide former.
153	90Ni/10Co	21.5	4	68	20	10	74	DLB-1,2	4	0	96	13	62	62/38	77	New higher surface area anode (0.5 m ² /g)
154	90Ni/10Co	21.5	4	67	20	10.5	74	DLB-1,2	4	0	96	13	64	50/50	77	Electrolyte composition test.
155	90Ni/10Co	21.5	4	68	20	10.5	74	DLB-1,2	4	0	96	13	63	68/32	77	Electrolyte composition test.
BENCH - SCALE																
7-35	90Ni/10Co	26	-	65	15	6.2	80	DLB-1,2	4	0	96	13	62	62/38	64	State-of-the-art performance of Ni/Co anode in conjunction with Ni current collector.
7-36	90Ni/10Co	28	5	65	16	9.5	72	LB-5	6	89	5	6	62	50/50	72	Endurance test of Ni/Co anode.
7-38	Ni	25	-	65	22	6.2	79	LB-5	6	89	5	6	62	50/50	73	Endurance test of a Ni anode stabilized by special treatment.

APPENDIX
TABLE B
SUMMARY OF CELL PERFORMANCE

CELL NO.	TOTAL* ENDURANCE, Hours	PEAK PERFORMANCE (at 115 mA/cm ²) mV	RESIS-TIVITY $\Omega \cdot \text{cm}^2$	WET SEAL EFFICIENCIES, %		COMMENTS
				ANODE	CATHODE	
<u>L A B - S C A L E</u>						
149	1500	860	0.56	95	100	Very good initial performance of 860 mV due to optimum electrolyte fill of anode, but final performance level stabilized at ~770 mV at 115 mA/cm ² .
150	590	622	0.72	96	100	Thin cathode cell was initially flooded due to too small MPS of cathode (as evidenced by high cathode gains), causing corrosion of cathode current collector, resulting in increased cell resistance.
-151	1500	827	0.55	96	100	The initial performance was lower than that of Cell 149, but it stabilized at a slightly higher final level (~790 mV).
152	1000	850	0.54	97	99	This cell containing a nickel anode alloyed with Yttrium displayed excellent performance for ~700 hours, then stabilized at a level comparable to Cells 149 and 151 (~780 mV at 115 mA/cm ²).
153	(865)	880	0.50	96	99	New higher surface area anode exhibited better performance (compare to Cell 149). Test is continuing.
154	(850)	880	0.47	97	98	To date, performance stability for this 50Li/50K, 64% liquid tile is better than for the 62Li/38K, 62% liquid tile (Cell 153). Test is continuing.
155	-	-	-	-	-	Planned to begin next quarter.
<u>B E N C H - S C A L E</u>						
7-35	(225)	900	0.52	100	99	State-of-the-art performance, Ni/10Co anode. After atmospheric tests were complete, cell was pressurized as Cell P7-2 (see Task 3).
7-36	(3475)	734	1.00	82	100	Endurance cell was initially flooded, later improved and has been stable for ~3000 hours.
7-38	(2187)	865	0.82	99	99	Specially treated Ni-anode has shown good performance stability beyond 2000 hours of endurance testing.

* Parentheses indicate test is continuing.

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