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DETERMINATION OF OPTIMUM ELECTROLYTE COMPOSITION
FOR MOLTEN CARBONATE FUEL CELLS

QUARTERLY TECHNICAL PROGRESS REPORT

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Prepared by

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MASTER

NOTICE

TITLE: DETERMINATION OF OPTIMUM ELECTROLYTE COMPOSITION
                    FOR MOLTEN CARBONATE FUEL CELL

CONTRACT NO.:   DE-AC21-86MC23264

CONTRACTOR: Energy Research Corp.
                    3 Great Pasture Road
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CONTRACT PERIOD: October 1, 1986 to September 30, 1988

OBJECTIVE: The objective of this study is to determine the
optimum electrolyte composition for molten carbonate fuel
cells. To accomplish this, the contractor will provide:

1. Comprehensive reports of on-going efforts to optimize
    carbonate composition.

2. A list of characteristics affected by electrolyte composi-
    tion variations; e.g. ionic conductivity, vapor pressure,
    melting range, gas solubility, exchange current densities
    on NiO, corrosion and cathode dissolution effects.

3. Assessment of the overall effects that these character-
    istics have on stateof-the-art cell voltage and lifetime.

CONTRACT TASKS:

Task 1. Preparation of Management Plan.

Task 2. Literature Survey to review completed and ongoing
         efforts to optimize MCFC electrolyte composition.

Task 3. Electrolyte Composition Screening to define "Optimum"
         in terms of carbonate properties and fuel cell para-
         meters.

Task 4. Test Plan submission describing methods to be used
         for identifying and verifying the optimum composition
         criteria.

Task 5. Experimental Determination of Optimum Electrolytes by
         a variety of out-of-cell and in-cell tests.

PROGRESS BY TASK

TASK 2 <u>LITERATURE SURVEY</u>

The minor effort under this task to continue surveying the literature for relevant information pertaining to composition effects on MCFC performance and material endurance have resulted in the folowing items. The basicity of alkali carbonate mixtures at $910^{\circ}C$ has been measured by Shores[66] with an electrochemical cell measuring $O^{=}$ ion activity. The experimental values were found to agree with previously calculated ones. This paper thus proves that Na content can effectively increase melt basicity, which in turn should decrease NiO solubility.

Uchida and Nishina[67] performed a thermodynamic analysis of alkali carbonate mixtures and proposed that the superoxide mechanism is predominant in Li/K and Na/K eutectic melts whereas the peroxide mechanism is dominant in the Li/Na eutectic melt. In the ternary eutectic melt, oxygen reduction is under mixed control of superoxide and peroxide paths. This conclusion is agreed upon by many researchers, as discussed in the Topical Report submitted in April, 1987.

Iwase, et al.[68] have also used the AC-impedance method to study a porous NiO electrode filled with different amounts of Li/K electrolyte. In this work they have demonstrated the feasibility of using the technique for cathode performance studies. Also, since it is shown that variation in electrolyte fill-level has a definite effect on performance, careful electrolyte impregnation and post-test fill-level determinations are planned for our experiments to be certain that only effects of electrolyte composition are measured.

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Ogawa et al.(69) have studied the effect of alkaline earth additives (Mg, Ca, Sr, Ba carbonates) on the dissolution behavior of NiO cathodes in $10\text{-}cm^2$, lab-scale fuel cells. The amount of NiO-dissolution was found to decrease by a factor of 1/2 to 1/3 with 5 mole% additions of $CaCO_3$, $SrCO_3$, and $BaCO_3$, which is roughly in agreement with most of ERC's findings. Furthermore, the NiO loss was found to be proportional to the period of operation and to the partial pressure of CO_2 in the oxidant, also in agreement with ERC's findings. The above results therefore confirm the general approach of using alkaline earth additives to suppress NiO dissolution. However, the effect of the different additives (i.e. Ca, Sr, Ba carbonate) could not be accurately differentiated, probably due to the usual difficulties with sampling and ppm. analytical techniques. Also, Ni transport rate and the mode of re-precipitation or crystallization toward the anode may be just as important as the NiO equilibrium solubility itself. Therefore, equilibrium solubility alone may not be entirely indicative of overall cathode loss rate or the ultimate effect it has on fuel cell performance.

Sato, et al. (70) have studied the interaction between Li/K melts and $LiAlO_2$ and have found that both stable and metastable solid carbonate phases exist. Furthermore, the melting points of these phases are affected by the presence or absence of $LiAlO_2$. Therefore, the Li/K carbonate phase diagram appears more complicated than what was originally thought. This phenomenon may influence the start-up or thermal-cycle behavior of cells and stacks and may, in particular, have to be taken into account with stack testing.

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TASK 3   <u>ELECTROLYTE COMPOSITION SCREENING</u>

This task, which was completed last August following the literature search and data evaluation phase of the work, resulted in a master table which lists the most important electrolyte properties that were then considered to have a definite effect on fuel cell performance. However, the detailed information and precise quantitative data for accurately comparing performance and endurance behavior were for the most part not yet available. At this time with more data, the table can be updated especially with respect to the carbonate characteristics that are of primary importance: i.e. NiO-solubility which affects cathode losses and 310S corrosion which affects long-term hardware stability. What follows is a review of this updated master table in connection with the Interim Program Evaluation which is a requirement in our work schedule at about this time.

3.1   INTERIM EVALUATION

This section presents the revised summary of the 8 candidate electrolyte properties with respect to the most important fuel cell characteristics to be considered. Discussion of the detailed data for Table 3.1 and the derived out-of-cell correlations that have been used will be presented under Task 5. The melting points and conductivity differences between candidate melts have not greatly changed. Additional data from recent literature has confirmed these values and except for some slight changes that might occur in the estimated values when a small amount of additive is present, these properties are not in doubt.

All candidate compositions except for the high lithium, off-eutectic, Li/K mixture (S_L) have melting points at least 100^0C below the MCFC operating temperature. The high lithium S_L composition has therefore a disadvantage here which could have some effect on uniform carbonate distribution upon start-up. In connection with cell resistance, it is clear that the slightly off- eutectic sodium blend has an advantage in providing up to 60% higher ionic conductivity than the present standard blends. One of the advantages for the sodium blend (N_L), therefore, is that it may lower the electrolyte contribution to a fuel cell's internal resistances both in the electrodes and in the matrix.

Theoretical predictions of electrolyte basicity affecting NiO-solubility have been confirmed by sets of duplicate nickel solubility measurements, as will be further discussed. Measured solubilities for each electrolyte were then correlated using a cathode, standard-rate-of-loss, conversion that has been derived from recently documented cells. From the latter values shown in Table 3.1, it is evident that the sodium blend (i.e. N_L, with the most alkaline electrolyte composition) should also be the most effective in limiting cathode electrode loss. By calculation, it may be estimated that the cumulative loss in 40,000 hours of operation with the sodium containing electrolyte will be ~52 mg/cm^2 for a typical cathode of nominally 200 mg/cm^2 nickel oxide. Hence, about 25% of original cathode weight could be transfered, which as far as our limited experience goes is acceptable for such an extended period of operation.

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TABLE 3.1

COMPARISON OF 5 CANDIDATE ELECTROLYTE COMPOSITIONS FOR OPTIMIZATION OF FUEL CELL CHARACTERISTICS

CODE	TYPE	Mol % COMPOSITION Li/Na/K	MELTING POINT °C	CONDUCTIVITY @ 650°C $(\Omega.cm)^{-1}$	CATHODE NiO LOSS RATE ** $\mu g/hr.cm^2$	310-S METAL LOSS $\mu m/(yr)^{1/2}$	OVERALL CATHODE IMPEDANCE $\Omega.cm^2$	ELECTROLYTE VAPOR LOSS ⟂ $\mu g/hr.cm^2$	ELECTROLYTE MIGRATION	γ-LiAlO2 POST-TEST STABILITY BET, m^2/g
S	STANDARD Li/K BINARY EUTECTIC-2	62/--/38	490	1.4	3.5	32	*	0.57	*	5*
S_L	Li/K BINARY HI LITHIUM, OFF-EUTECTIC	72/--/28	575	1.7	2.0	28	*	0.49	Li/K mobility equal	5*
N_L	Li/Na BINARY SLIGHTLY OFF-EUTECTIC	60/40/--	540	2.3	1.3	29	*	0.12	*	*
S_M	Li/K BINARY + ADDITIVE (0.5 Mg)	62/--/38	490*	1.4 *	1.7	31	*	0.57	*	*
S_X	Li/K BINARY + ADDITIVE (5 Ba)	59/--/36	>490 <510	1.4 *	2.5	32	*	0.55	*	*
	ELECTROLYTE SELECTION GOALS		<550	>2.0	<3.5	<<50 or <3 mil/ 40,000 hr	<0.9	<<0.5 (esp. in fuel at- mosphere)	<10 % Change In Cation Ratio	>5

* Estimated Value or Work in Progress: ⟂ Modeled and Calculated by the MOH and K-vaporization mechanisms.
** Using Standard ERC Correlation for in-cell Cathode Losses vs. Measured Solubility at a typical CO2 concentration of 13% in Oxidant Gas.

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In connection with corrosion, the major concern of greater hardware attack in the presence of sodium containing blends has been proven incorrect as far as our present work is concerned. When comparing the amount of attack on the superior 310S type of stainless steel used, there is very little variation in corrosion due to the different candidate electrolyte compositions. If anything can be concluded, it is that the 60Li/40Na blend, N_L, even shows a somewhat lower potential for corrosion than the standard 62Li/38K carbonate composition. On the other hand none of the candidate electrolytes, even when tested up to 3000 hours using accelerated exposure with higher than usual temperature and humidity conditions, have resulted in any kind of serious corrosion projections. For the N_L blend, the low metal loss rate noted of 29 $\mu m/(yr)^{\frac{1}{2}}$ translates to approximately 2.4 mil of metal loss due to corrosion in 40,000 hours which is probably acceptable for hardware materials of average 10 mil thickness. Furthermore, the maximum corrosion penetration into the steel at a few sites is a median of somewhat less than 3.5X the average metal loss depth and thus catastrophic leakage across any gas separating membrane also appears unlikely.

Thus, the fact that melt composition does not strongly bear on corrosion differences, allows this primary factor for optimization to be eliminated, making NiO dissolution the major selection criterion. From this summary of recent data, it can therefore be concluded that a definite optimization of primary electrolyte characteristics can be achieved. The best candidate mixture for the upcoming bench-scale phase of testing is therefore the N_L blend with the other composition being the standard comparison electrolyte. Eventhough not all of the out-of-cell experiments are complete, it has been necessary for the present schedule to make this decision

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because a good deal of preparatory work (e.g. electrolyte
filling of the components for the first set of four
bench-scale cells) is already in progress. This important
selection has therefore been made in as much as adequate
corrosion behavior as well as the lowest NiO solubility were
found with this melt. Other advantages which also contributed
to this decision were that the N_L blend has the highest
conductivity of any of the candidate compositions and, in the
absence of potassium carbonate, the melt will have lower
electrolyte volatility on the anode side of the cell. Lower
ohmic loss and therefore a somewhat better overall performance
and better performance endurance due to less electrolyte being
transfered are the additional expected benefits for this melt
over the standard 62Li/38K carbonate mixture.

Although alkaline-earth additives such as $MgCO_3$ reduce
the NiO-solubility to some extent, this does not provide a
very large advangtage for the S_M blend which has otherwise
nothing special to add. There is also the concern that the
very small quantity of $MgCO_3$ which can only dissolve in a
carbonate melt will easily deplete and hence may only have a
short-term effect. Moreover, it has been found in recent ERC
bench-scale cells that the recrystallizaton habit of
transferred nickel being reduced in the matrix can be quite
unusual when the electrolyte contains $BaCO_3$ additive (i.e.
dendritic shaped nickel crystals appear to form). This could
eventually cause cell short-circuiting across the matrix and
hence appears to argue against the use of additives for the
present. Some thought is being given, however, to combining
magnesium carbonate with sodium-containing blends to possibly
further suppress NiO-solubility in the already optimized
composition. Such a test might be recommended for the 5000
hour bench-scale cells to be operated when the present
1000-hour tests are complete.

TASK 5   EXPERIMENTAL DETERMINATION OF ELECTROLYTE PROPERTIES

During this quarter, corrosion testing of 310S stainless steel in the 5 candidate electrolytes was completed. Coupons were cross-sectioned and metallographically examined for metal loss rates and maximum corrosion penetration. The NiO-solubility study which consists of high precision analytical determinations of dissolved nickel in the candidate melts is also essentially finished. Cathode performance and LiAlO2-stability experiments are in progress, while the electrolyte migration experiments and bench-scale cell testing are being prepared.

5.1   CORROSION

Corrosion testing of 310S stainless steel coupons for 500 and 3000 hour periods in the five candidate electrolytes has been completed. A number of conclusions from observations and micrographs were reported in the October and November monthly reports which will be summarized below.   The treatment of post-test coupons by washing and cross-sectioning for metallographic analysis has already been described in detail in earlier reports.

The corrosion rate of 310S in the five candidate melts were measured by high definition optical microscopy and compared in terms of both the maximum depth of corrosion attack and the average amount of metal loss with respect to the original coupon surface.   Since these measurements vary depending on the location of corrosion attack upon the specimen surface, median rates were determined by averaging the general corrosion depths and metal losses at many points on the coupons. It is suggested that the non-uniform corrosion is caused primarily by the originally non-uniform distribution of carbonate on the coupons' surfaces and possibly also by shielding of areas under the electrode plaque where the electrolyte is initially stored inside the U-bend coupons.

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Summary Figures 5.1 and 5.2 show that this average corrosion and maximum depth of attack due to molten alkali carbonate on 310S stainless steel results in only minor differences between the five candidate melt compositions. This holds both after 500 and 3000 hours of duplicate tests under exactly the same temperature and humidity conditions ($700^{\circ}C$ and 5% H_2O). Significantly, these differences between the melts, in fact, should be small if corrosion and the subsequent stability of the protective scales are primarily a function of melt alkalinity. This will be the case when the solubilities of $LiFeO_2$ and Li_2CrO_4 — i.e. the major constituents of the protective outer scales on stainless steel[72] — are the main determining factors for scale stability. The oxides exist in equilibrium with their corresponding ions in solution, such as Li^+, Fe^{3+}, Cr^{6+}, $O^=$, etc. Hence solubility must clearly be a function of the local metal ion, and also the oxygen ion ($O^=$), concentrations. Thus, electrolyte basicity in terms of $O^=$ at the metal surface is an important parameter.

However, it may be assumed because of the generally low absolute magnitude of $O^=$ ion concentration in carbonate electrolyte, that the slight differences in basicity which occur between the different melt compositions are probably insufficient to cause a major variation in corrosion attack. The average metal loss and maximum attack depth due to corrosion are seen to be rather insensitive to melt basicity; for the cross-sections of the 5 candidate melts over a 500 hour period these parameters are approximately 7.4 and 30 μm. If corrosion rates may be assumed to be parabolic with time, this represents a general metal loss rate of 31 $\mu m/(yr)^{\frac{1}{2}}$. Similarly, average metal losses and maximum depth of attack found in the 3000 hour tests are approximately 18 and 58 μm, respectively. This translates into a general metal loss rate of 30.7 $\mu m/(yr)^{\frac{1}{2}}$, which is very close to that previously calculated for the 500 hour period of testing. The assumed parabolic corrosion behavior is thus also confirmed from these tests.
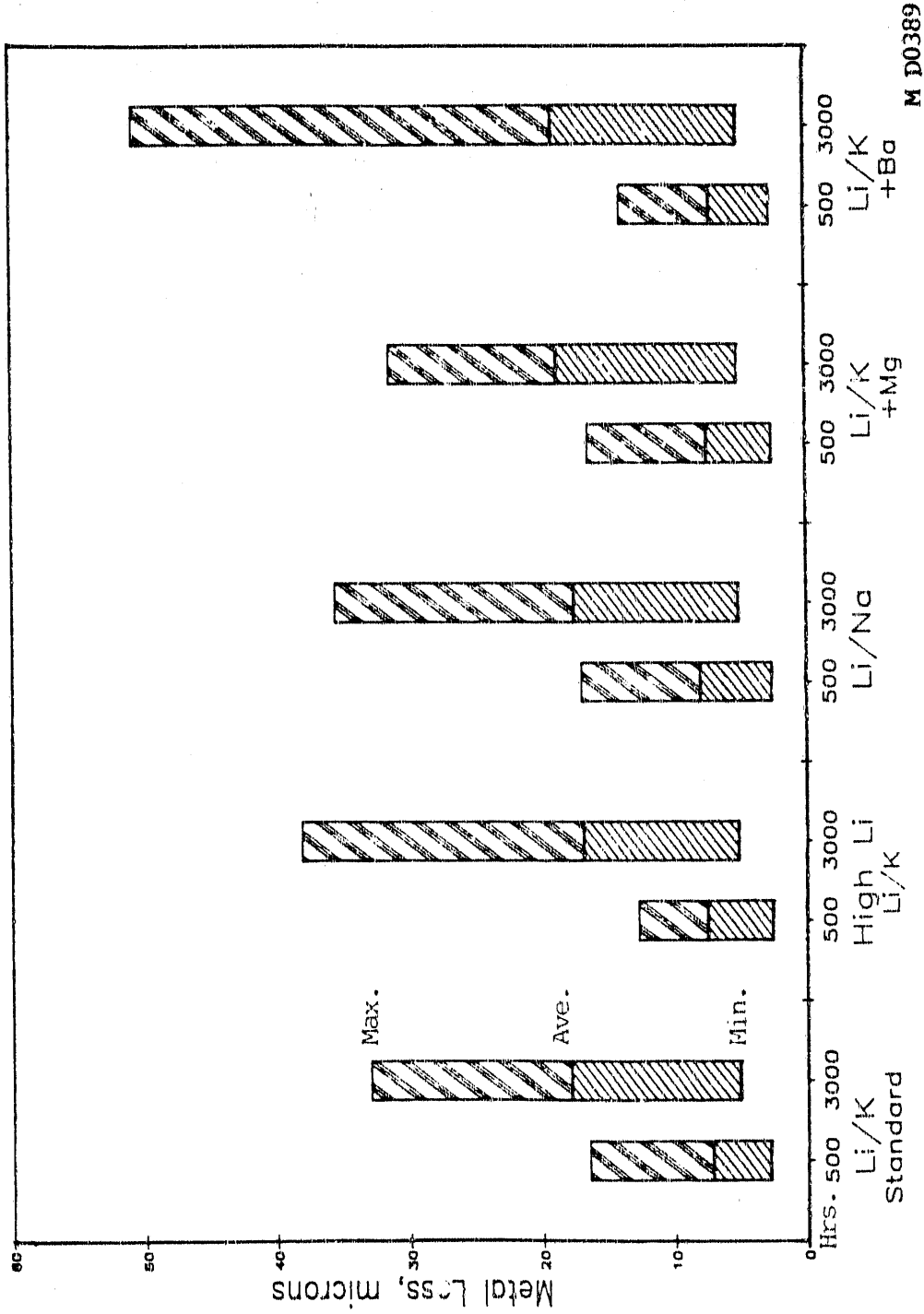
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FIGURE 5.1 COMPARISON OF 310S STAINLESS STEEL CORROSION IN 5 CANDIDATE ELECTROLYTES
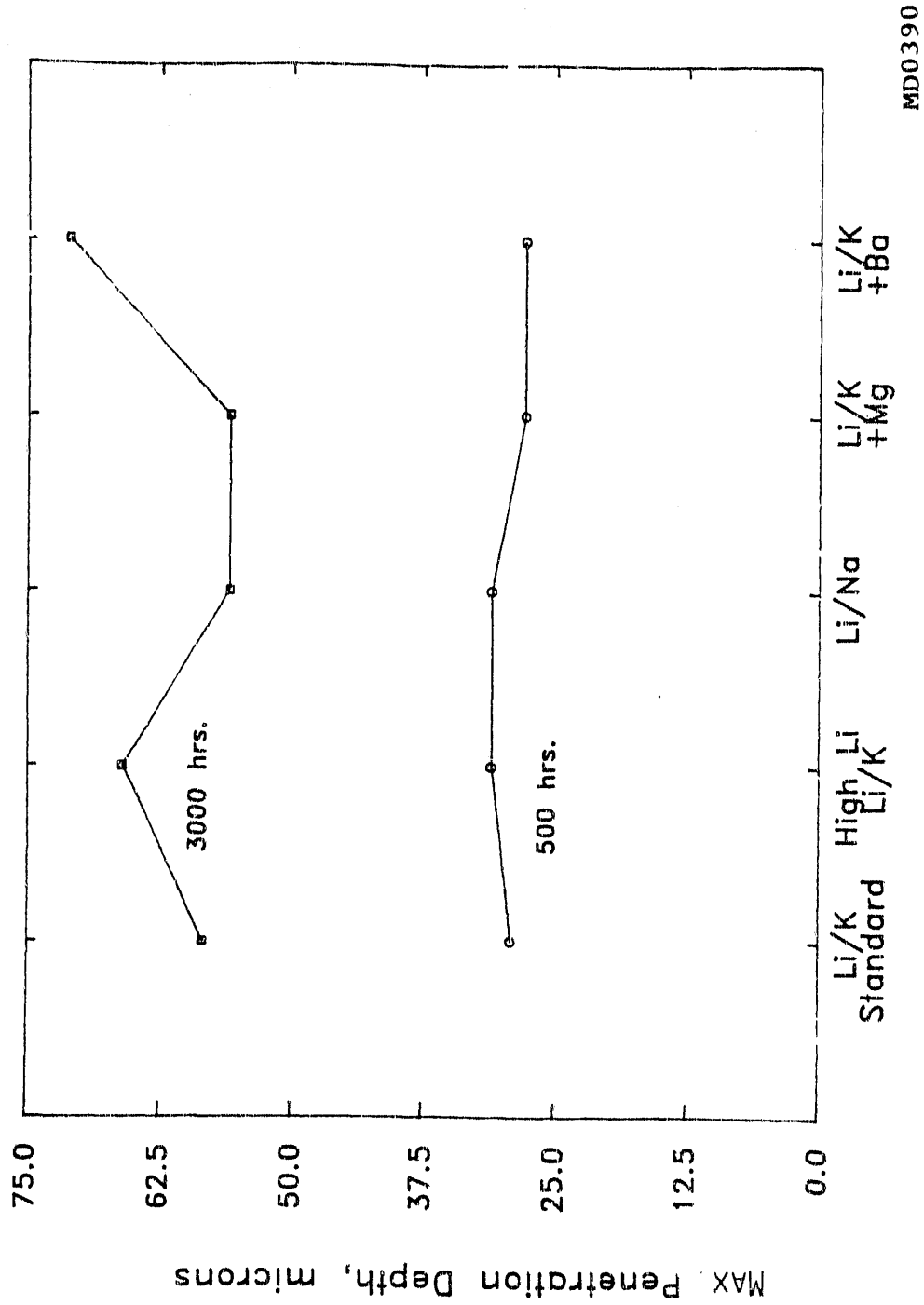(corrosion in different locations of the specimen)

MD0390

FIGURE 5.2   COMPARISON OF 310S STAINLESS STEEL CORROSION: MAXIMUM
PENETRATION IN 5 CANDIDATE ELECTROLYTES

In general, it appears that the scale on the top surfaces of the cross-sections (i.e. facing the inside of the U-bend coupons) are slightly thicker in the case of the sodium melt, but when considering the bottom surfaces they are thicker in the case of the potassium melt (i.e. when facing outward from the U-bend). Maximum attack depth into the metal such as penetration along the grain boundaries also varies a great deal with location and sometimes appears to be slightly greater with the sodium blend. However, as illustrated in the micrographs of corrosion scales formed on two typical specimen cross-sections obtained with 62Li/38K and 60Li/40Na melts after 3000 hours of testing (cf. Figure 5.3), these depths of attack showing the maximum differences in oxide penetration are rather insignificant.

This now completes the corrosion testing for this program. The fact that melt composition does not appear to significantly affect corrosion with respect to the superior 310S alloy, therefore, allows the elimination of one previously perceived important factor in electrolyte optimization, leaving NiO dissolution the only primary screening test.

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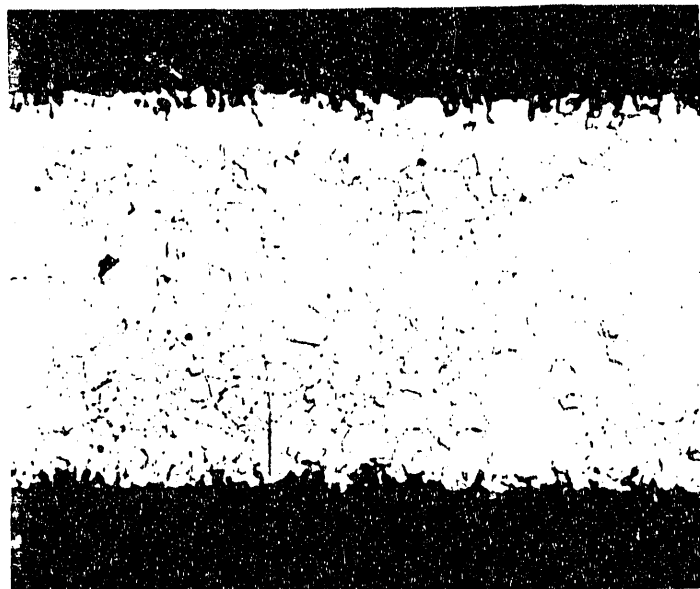
(62Li/38K)CO$_3$

50 µm
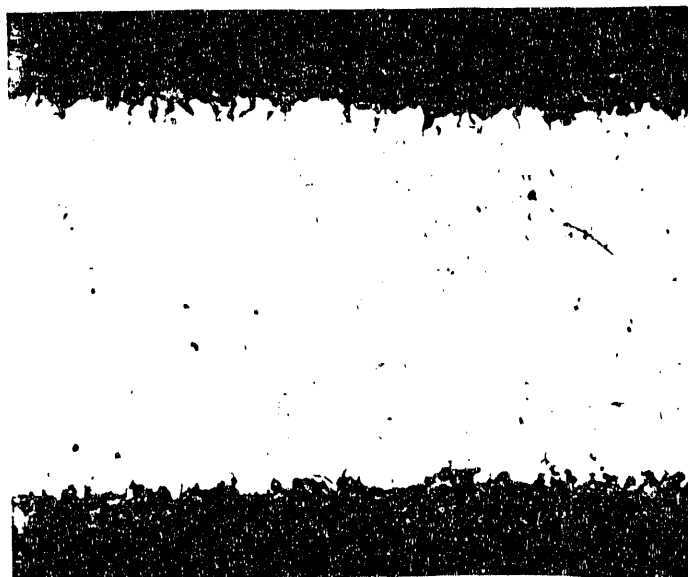
(60Li/40Na)CO$_3$

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FIGURE 5.3 TYPICAL CORROSION OF 310S IN TWO ELECTROLYTES
(700°C, 3000 hours)

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5.2 NiO DISSOLUTION

During this reporting period, NiO-dissolution experiments in 5 candidate melts (in duplicate) were also being completed. The melt samples from the pottests, after dissolution in HCl, were all analyzed by the high sensitivity technique of Atomic Emission Spectroscopy using an Inductively Coupled Plasma (ICP) to excite the spectral lines. The NiO solubilities were accentuated by performing two test series using an 85% CO_2 atmosphere with results as illustrated in Table 5.1. The solubility measured in the 72Li/28K melt (S_L) shows that a substantial improvement may be obtained over the standard melt (S) by an increase in the more alkaline lithium content of a melt. This is about equivalent to the small alkaline earth additions that were also tested, but does not equal the very low solubility obtainable in 60Li/40Na melt (N_L). According to these results, NiO solubility decreases in the sequence $S > S_X > S_L > S_M > N_L$. It is interesting to note that the addition of $BaCO_3$, even at a much higher solution level, is not as effective in suppressing NiO dissolution as the addition of $MgCO_3$. This is consistent with the salt dissociation constants which show that the latter additive is the more basic alkaline earth carbonate.

TABLE 5.1

NiO SOLUBILITY OF CANDIDATE MELTS AT 650°C

Code	Electrolyte	Solubility in 85% CO_2-Atmosphere (wppm)	
		Test Series No.1	Test Series No.2
S	62Li/38K	19 ± 4	20 ± 5
S_L	72Li/28K	11 ± 3	10 ± 2
N_L	60Li/40Na	8 ± 1	6 ± 1
S_M	Li/K + 0.5Mg	10 ± 2	8 ± 2
S_X	Li/K + 5 Ba	16 ± 3	12 ± 2

5.3 CATHODE PERFORMANCE

Preliminary results reported earlier showed that a large
low-frequency loop existed which was apparently caused by some
high impedance, mass transfer process. Such results were not
consistent with what has been reported(16), and was felt to be
due to configurational problems of poor gas access to the NiO-
electrode, difficulties with non-uniform temperature distribu-
tion in the furnace, and also uneven electrolyte distribution
between the two NiO-electrodes in the test package. There-
fore, the test configuration was modified to allow better gas
access to the measured cathodes and a flexible thermocouple
was inserted in the upper gas distribution/current collector
for more accurate control of the temperature.

It has also been frequently reported that the level of
electrolyte fill has a strong effect on cathode performance.
Hence uniform and sufficient electrolyte distribution is an
important factor to insure accuracy in this type of study. The
initial cathode fill-levels used were 25% of electrode poro-
sity, which may have been too low as a result of the consi-
derable electrolyte creepage as well as some of the corrosion
which appeared to have occurred. Future experiments will
start with carefully controlled, uniformly distributed, 40%
carbonate-impregnated, NiO cathodes.

5.4 LiAlO₂ STABILITY

Two duplicate sets of LiAlO₂-stability tests, being
obtained in both oxidant and in fuel atmosphere for three
candidate melts, were started during the last quarter period
and have now been completed. The electrolyte matrix tapes used
are of standard ERC composition and are filled in-situ with
the candidate electrolytes after the organic binders are
burned out in the test chamber in the same manner as would
happen during start-up of a fuel cell.

The electrolyte quantities that fill these matrices are initially always contained in the electrodes which contact the tape. A number of the test samples, together with the original LiAlO2 powder, a dry burned-out tape, and a 0-hour carbonate-filled sample, have been sent to Foote Minerals Company which is assisting with the complete characterization and analysis of the LiAlO2 matrix materials.

5.5   ELECTROLYTE MIGRATION

Initial experiments for the electrolyte migration testing are in progress. The two alumina blocks which compress a gasket sample within the test rig have been cut and holes have been drilled in the end sections. A standard fibrous ZrO2 gasket, impregnated with ZrO2-powder to reduce porosity, is being used in these migration studies. For initial testing, the filled ZrO2 gasket is sandwiched between the two alumina blocks and compressed to a final thickness of 30 mils (from an initial thickness of ~50 mils). Carbonate powder is then heaped around the gasket and the whole assembly is pre-treated at $650^{\circ}C$ to allow the carbonate to melt and completely absorb in the porous gasket. The assembly is now being tested with different melts for evaluation of this filling procedure. Actual experimental runs are expected to start during the next reporting period.

5.6   IN-CELL TESTING

All Ni/Al anode and Ni cathode plaques as well as LiAlO2 matrix tapes for the eight bench-scale cell tests have been fabricated. The 310S (cathode) and 316L (anode) cell frames have been machined and the inside of the anode frames have been electrolytically nickel-plated. Some unexpected difficulties were initially encountered with the plating in that the coatings were too thin and of poor quality. These coatings have now been stripped and re-plating as well as all other surface treatments for wet seal protection, etc. are now being completed.

Test stations are also being modified to replace the former on-off temperature controllers by PID controllers which apply the heater currents in a more even manner. This development is expected to substantially reduce thermal distortion in the cell's end frames during operation and thus eliminate excessive load fluctuations which in the past were caused by a loss of contact. This improvement is expected to provide a more consistent picture of instantaneous cell behavior. An additional safety device has also been installed at each test station allowing all cells to be simultaneously put on OCV when unexpected problems occur such as any kind of power outage or gas supply breakdown even if it could cause just one of the cells to run at an excessively low voltage. Thus, it will guarantee that all cells at all times are operated under exactly the same regime of load or open circuit conditions in case any problems occur at night or over the weekends. Multipoint recording is also being used to constantly monitor cell voltage, current, temperatures on all cells, preheaters and humidifiers, and also pressure drops on all four cells.

Single cell testing with the 62Li/38K and 60Li/40Na electrolytes will be started as soon as all final preparatory steps, quality control and the precision assembly of the cells is completed.

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