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Research and Development Technical Report
ECOM- 0143-2

ELECTRODE-ELECTROLYTE INTERACTIONS

MAGNESIUM ANODES : : meta-DINITROBENZENE CATHODES

SECOND SEMI-ANNUAL REPORT

By

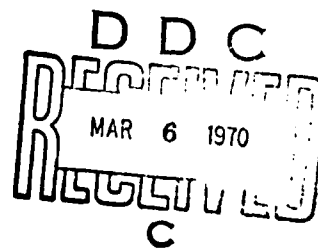
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DECEMBER 1969



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CHEMICAL ENGINEERING DEPARTMENT

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Electrode - Electrolyte Interactions
Magnesium Anodes : meta Dinitrobenzene Cathodes

1 July 1968 to 30 December 1968

Report No. 2

Contract No. DAAB07 - 68-C-0143

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Abstract

The electrochemical dissolution of magnesium (AZ-21) and the h.e.r. on magnesium (AZ-21) in magnesium perchlorate, sodium perchlorate and a mixed electrolyte of sodium and magnesium perchlorate has been investigated. In the sodium perchlorate electrolyte, 0.1M to 4.0M, the Tafel Slope of the h.e.r. was unaffected by the direction of the potential sweep. In the magnesium perchlorate electrolyte, 0.2M to 2.0M, the Tafel Slope of the h.e.r. changes with direction of the potential sweep. The anodic Tafel Slopes in the pure electrolytes was 50 mv and 30 mv in the mixed electrolytes.

The rate of magnesium dissolution in the magnesium perchlorate was about an order of magnitude faster than that in the sodium perchlorate electrolyte. On the other hand the corrosion current density of magnesium in the magnesium perchlorate was three (3) orders of magnitude greater than in the sodium perchlorate electrolyte.

Corrosion rates of magnesium in magnesium perchlorate and magnesium perchlorate saturated with magnesium hydroxide were also investigated. It was found that addition of the hydroxide reduced the corrosion rate by about 50%. It was also determined that magnesium did not exhibit the Negative Difference Effect.

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Introduction

Although the magnesium - meta dinitrobenzene battery is potentially very attractive, it has not been developed into a reliable energy source. Failure, low terminal voltage and low efficiency plague the system. A review of the literature⁽¹⁾ has indicated that data pertinent to the magnesium anode and the meta-dinitrobenzene cathode are few. In addition, some of these data are in question. To design a reliable cell a reasonable understanding of the anode and cathode reaction is necessary. Thus a systematic study of the electrochemical characteristics of these electrodes is under study.

Polarization Data

Experimental Procedure:

The electrochemical cell and the three electrode systems employed in this study were described previously⁽¹⁾. The test electrodes investigated were standard "A" magnesium (AZ-21). A constant surface area was obtained by immersing the open end of each battery can into molten wax. As masked, the wax-free end of the magnesium can provide a surface area of 14 cm^2 .

The cell was filled with the appropriate electrolyte of known pH. The active end of the magnesium "A" can was immersed into the

solution. The stable rest potential was recorded. Each run was initiated by holding the magnesium test electrode at the initial potential for 10 minutes. The taking of the potentiostatic polarization data was then initiated. At each potential value the current was measured after a fixed period of time, usually five (5) minutes. Potential increments of 25 mv were used.

In an attempt to obtain reproducible polarization data (or the magnesium alloy, AZ-21,) it was found that the potential from which the run was initiated, effected the polarization data. Thus, the effect of starting potential on the nature of these electrochemical reactions was investigated. For each experimental run a base potential, -1.70v to -1.90v SCE, was chosen. Again 25 mv increments were used. For each established base potential, in the given electrolytes, the runs were repeated at least once or until reproducible data were obtained. Both the anodic dissolution of magnesium and the h.e.r. on the magnesium alloy AZ-21 were studied. The pH of the electrolyte at the beginning and end of each run was recorded.

The electrolytes used in this study are listed in Table I.

Table I.

Electrolytes Investigated

Single Salt Electrolytes

2.0M $\text{Mg}(\text{ClO}_4)_2$

1.0M $\text{Mg}(\text{ClO}_4)_2$

0.6M $\text{Mg}(\text{ClO}_4)_2$

0.2M $\text{Mg}(\text{ClO}_4)_2$

4.0M NaClO_4

1.2M NaClO_4

0.4M NaClO_4

Mixed Salt Electrolytes

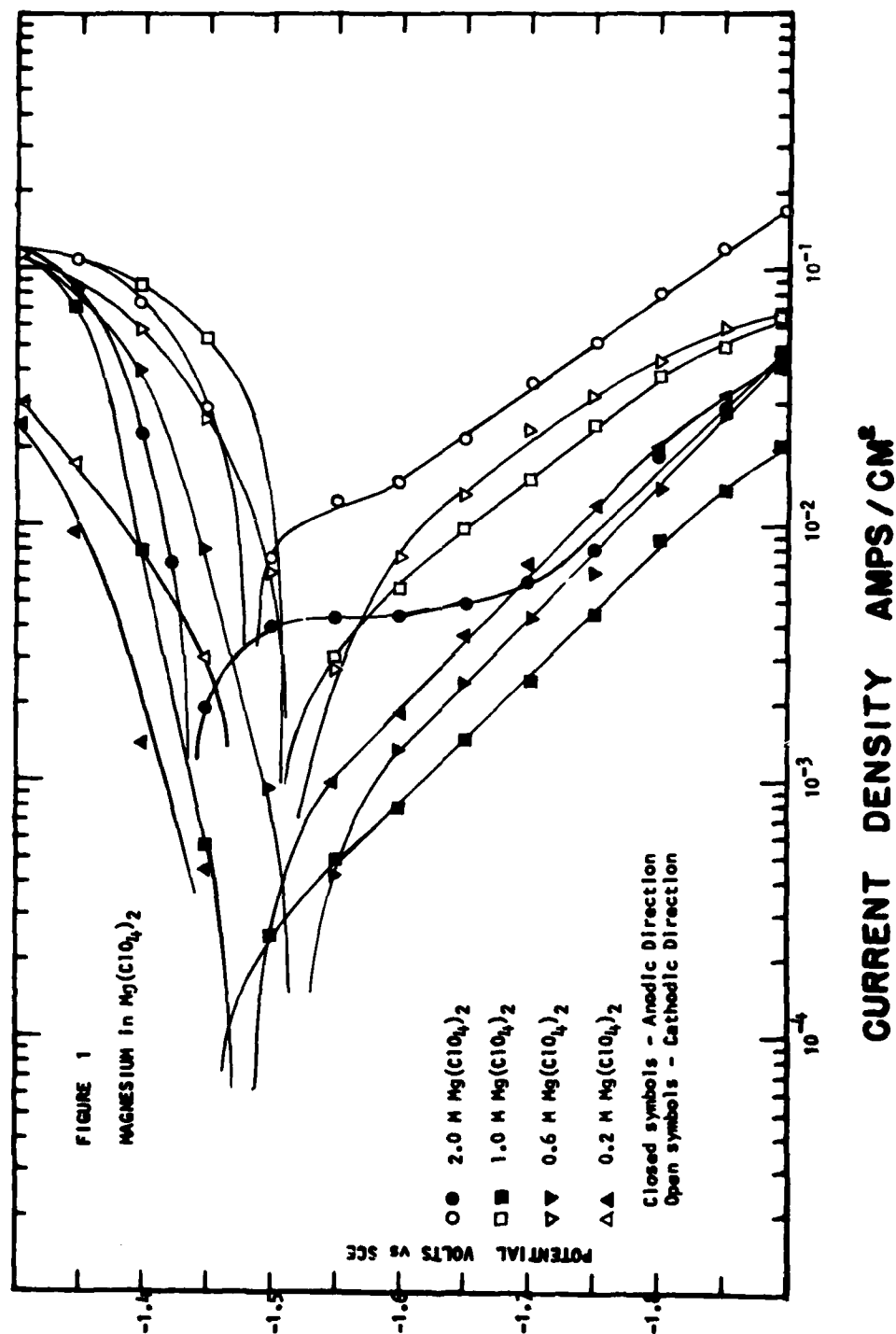
3.96M NaClO_4 - 0.02M $\text{Mg}(\text{ClO}_4)_2$

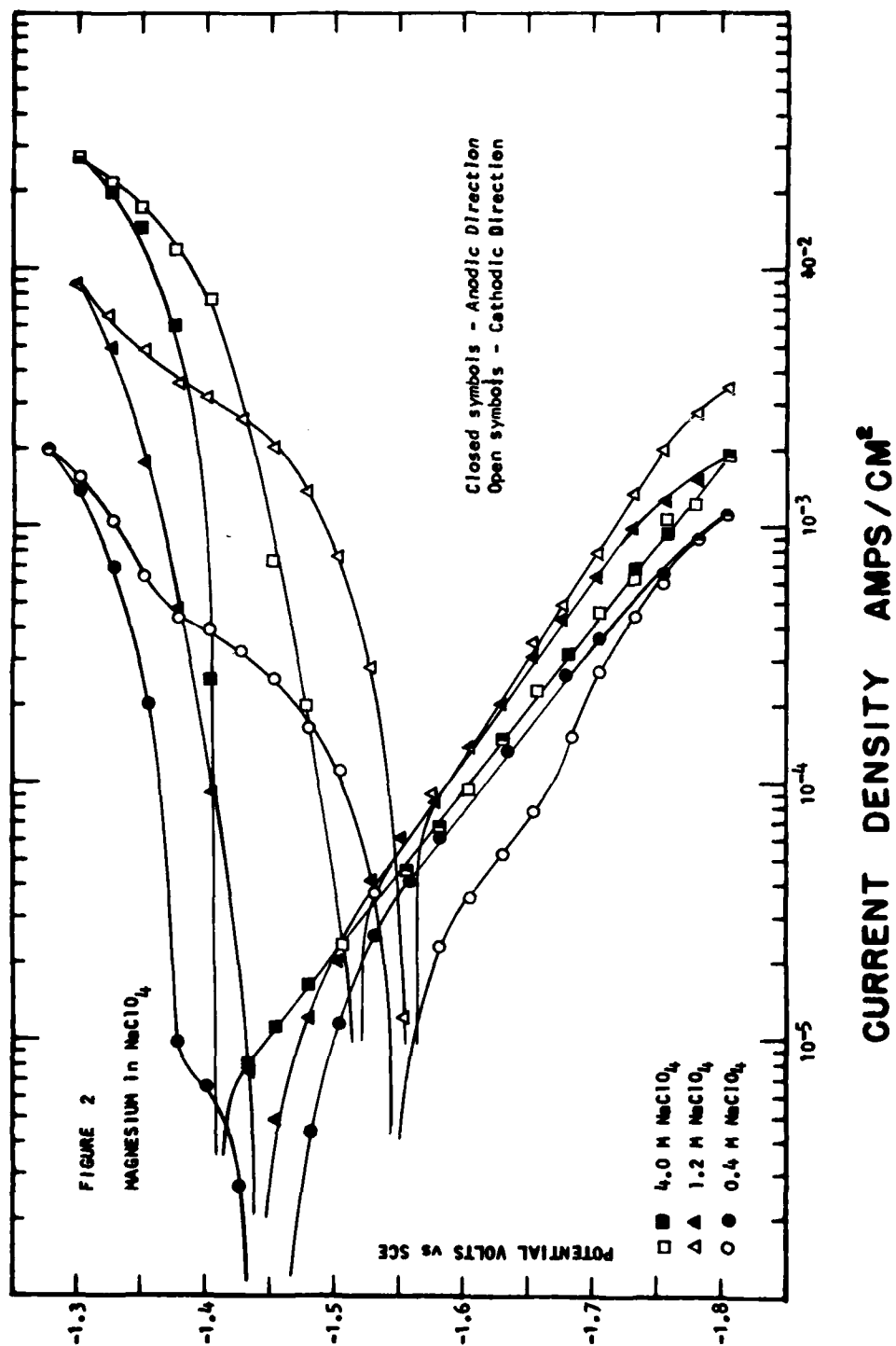
3.60M NaClO_4 - 0.2M $\text{Mg}(\text{ClO}_4)_2$

Results and Discussion:

Figure 1 shows the polarization data for the dissolution of magnesium and the h.e.r. on magnesium in $\text{Mg}(\text{ClO}_4)_2$ at concentrations of 0.2M, 0.6M, 1.0M, and 2.0M. The h.e.r. on magnesium had a Tafel slope of $200 \text{ mv} \pm 10 \text{ mv}$ with anodic potential steps and $270 \text{ mv} \pm 10 \text{ mv}$ with cathodic potential steps. The rate of the h.e.r. measured with anodic potential increments was about one order of magnitude slower than with cathodic potential increments. The anodic dissolution of the magnesium anode was not at all well defined. An anodic Tafel slope of approximately 50 mv is observed. The maximum dissolution rate of magnesium (AZ-21) in 0.6M, 1.0M and 2.0M $\text{Mg}(\text{ClO}_4)_2$ had the same value of about 140 ma/cm^2 at a potential of -1.30v vs SCE. The maximum dissolution rate of the magnesium (AZ-21) anode in the 0.2M $\text{Mg}(\text{ClO}_4)_2$ was about 30 ma/cm^2 at a potential of -1.30v vs SCE. Table 2 summarizes the data observed for the magnesium electrode in $\text{Mg}(\text{ClO}_4)_2$.

Figure 2 shows the polarization data for the dissolution of magnesium and the h.e.r. on magnesium in NaClO_4 at concentrations of 0.4M, 1.2M and 4.0M. The h.e.r. on magnesium had a Tafel slope of $170 \text{ mv} \pm 30 \text{ mv}$. The h.e.r. did not appear to be dependent on ionic





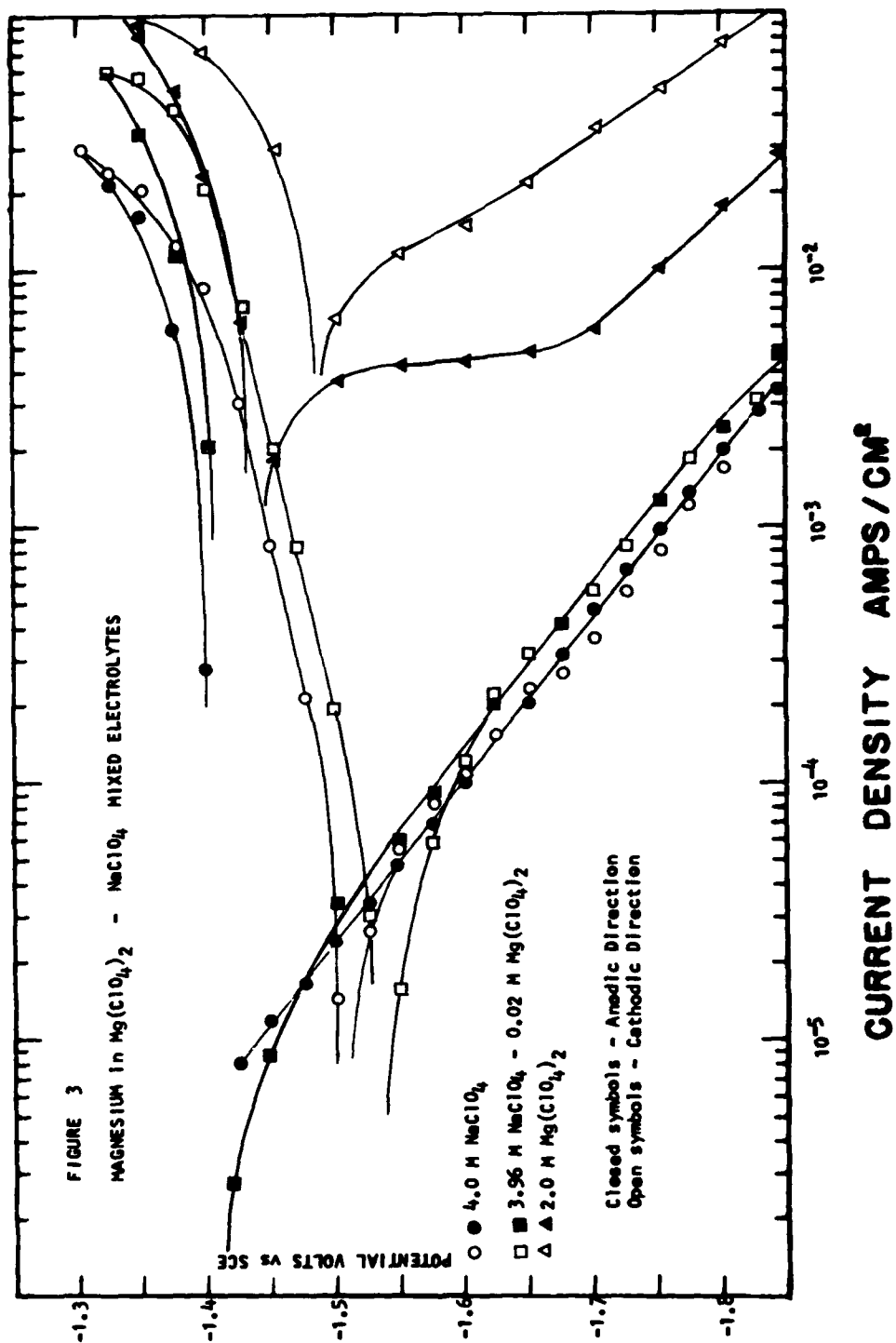
strength or the direction of the potential sweep. The maximum rate of magnesium dissolution in the NaClO_4 was proportional to ionic strength. An anodic Tafel slope of about 45 mv was observed. Considerable hysteresis was observed between the anodic and cathodic potential sweeps for the magnesium dissolution. Table 2 summarizes the data observed for the magnesium electrode in NaClO_4 .

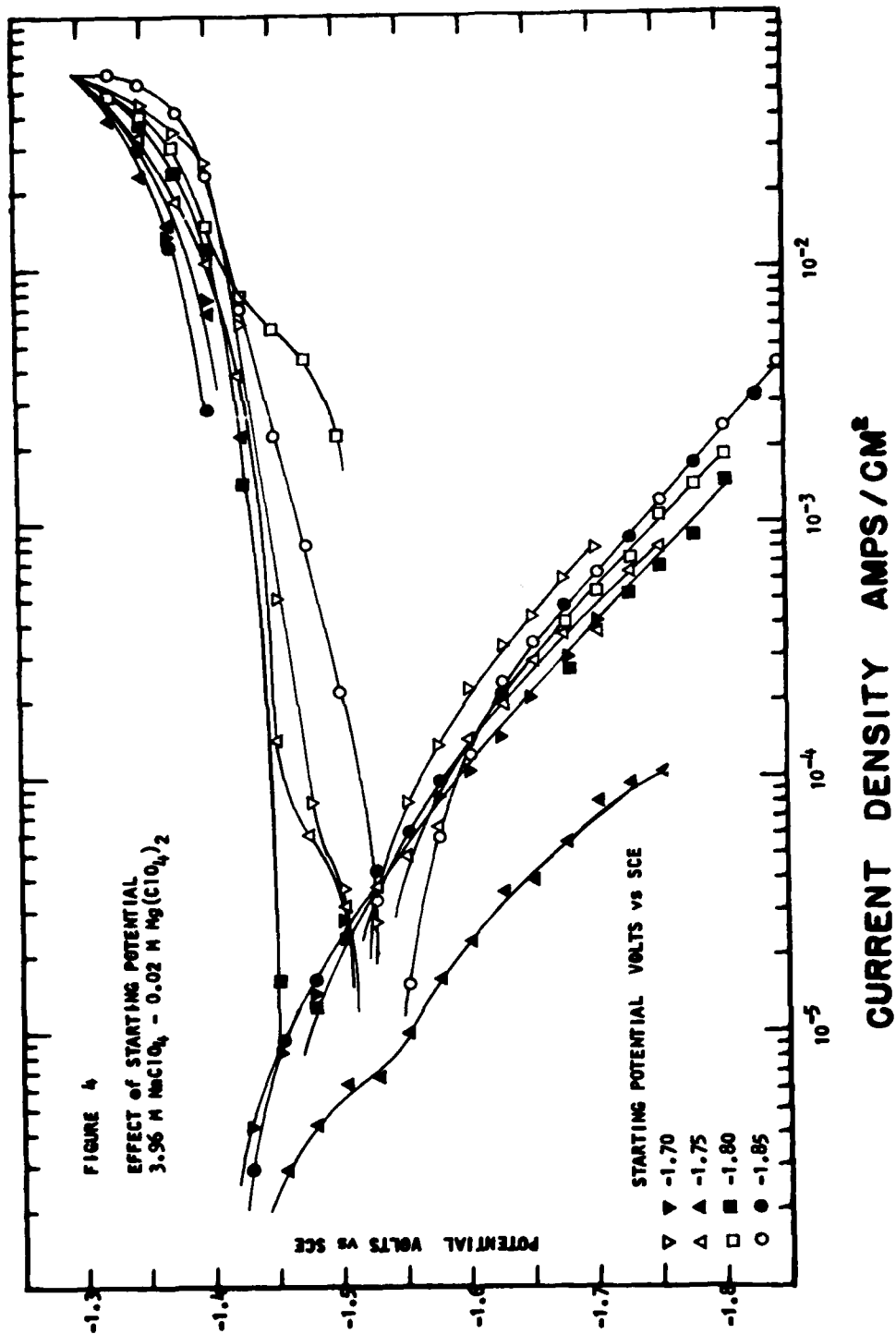
Figure 3 shows the comparison between the polarization for a magnesium electrode in 4.0M NaClO_4 and a magnesium electrode in a mixed electrolyte of 3.96M NaClO_4 and 0.02M $\text{Mg}(\text{ClO}_4)_2$. The h.e.r. reaction was unaffected by the $\text{Mg}(\text{ClO}_4)_2$, but the rate of magnesium dissolution was increased. The anodic and cathodic Tafel slopes also remained unchanged, but the corrosion current increased slightly. The electrochemical data for magnesium dissolution and h.e.r. on magnesium are summarized in Table 2.

In an attempt to obtain reproducible polarization data for the magnesium alloy AZ-21, it was found that the potential from which the run was initiated affected the data. Thus, the effect of starting potential on the nature of the electrochemical reactions was investigated. Figure 4 shows the polarization data for magnesium in 3.96M NaClO_4 - 0.02M $\text{Mg}(\text{ClO}_4)_2$ with starting potentials of -1.70v, -1.75v, -1.80v and -1.85v vs SCE. From these data it appears that a

Table 2
Electrochemical Parameters for Magnesium in Perchlorate Electrolyte

Electrolyte	pH Initial Final	Rest Potential v vs SCE	Starting Potential	Potential Current Cat./Ano.	H.E.R. Tafel Slope		Terminal Anodic Potential
					Anodic Sweep mv	Cathodic Sweep mv	
0.2M $Mg(ClO_4)_2$	8.0/2.5	-1.45	-1.90	-1.50	200	270	-1.275v
0.6M $Mg(ClO_4)_2$	8.4/8.0	-1.55	-1.90	-1.55	200	270	-1.225v
1.0M $Mg(ClO_4)_2$	7.6/7.8	-1.56	-1.90	-1.48	200	270	-1.325
2.0M $Mg(ClO_4)_2$			-1.90	-1.45	210	280	-1.225
0.4M $NaClO_4$	5.9/10.1	-1.43	-1.80	-1.45	200	UD	-1.275
1.2M $NaClO_4$	5.8/10.0	-1.42	-1.80	-1.43	150	130	-1.30
4.0M $NaClO_4$	5.4/10.1	-1.42	-1.90	-1.40	190	190	-1.30
3.96M $NaClO_4$							
0.02M $Mg(ClO_4)_2$	8.0/8.5	-1.425	-1.65	-1.42	180	180	-1.325
3.96M $NaClO_4$							
0.02M $Mg(ClO_4)_2$	6.2/8.6	-1.435	-1.70	-1.42	180	180	-1.30
3.96M $NaClO_4$							
0.02M $Mg(ClO_4)_2$	6.5/8.4	-1.435	-1.75	-1.43	180	180	-1.325
3.96M $NaClO_4$							
0.02M $Mg(ClO_4)_2$	6.3/8.6	-1.445	-1.80	-1.45	180	180	-1.30
3.96M $NaClO_4$							
0.02M $Mg(ClO_4)_2$	7.6/8.6	-1.435	-1.85	-1.48	180	180	-1.35





greater hysteresis occurred in the anodic portion of the curve when a more active starting potential was used. The Tafel slopes are unchanged but the transition potentials for current change become more noble with more active starting potentials. The polarization data, anodic direction, for an initial potential of -1.75 v vs SCE appear to be in error and are to be repeated. The electrochemical parameters for these data are also given in Table 2.

Current Efficiency and Corrosion

Experimental Procedure:

Current efficiency and corrosion rate tests were also performed with the magnesium alloy, AZ-21, anodes. Strips of the alloy, $1\text{ cm} \times 5\text{ cm}$ were used. The strips were cleaned with emery paper. In addition to cleaning with emery some of the electrodes were cleaned with acetic acid as described by P.F. King⁽²⁾. Subsequent to cleaning the magnesium strips were weighed with an analytical balance and immersed in the test vessel or connected in the dissolution circuitry.

At the conclusion of each run the magnesium strips were cleaned under running water with a brush dipped in dilute acetic acid. When heavy pitting occurred, the dissolution or corrosion powder was removed with physically great care. The electrode was then cleaned as indicated above with dilute acetic acid.

The duration of the runs were approximately 3-4 hours where the current range was 5-30 MA and approximately 20 hours where the range was 1-5 MA. The pH was obtained by means of a Beckman pH meter. As a matter of economic convenience, the same solutions were used throughout each series of runs.

The current was supplied by means of 12V automobile battery, regulated by means of variable resistance box and by means of a Simpson meter.

Visual corrosion tests were performed by placing a weighed strip of Mg in a sealed bottle filled with the electrolyte. After a certain amount of time the weight loss and visual observations were duly noted.

The dissolution and corrosion of magnesium, AZ-21, were studied in 2M $\text{Mg}(\text{C}_2\text{O}_4)_2$ and 2M $\text{Mg}(\text{C}_2\text{O}_4)_2$ saturated with $\text{Mg}(\text{OH})_2$.

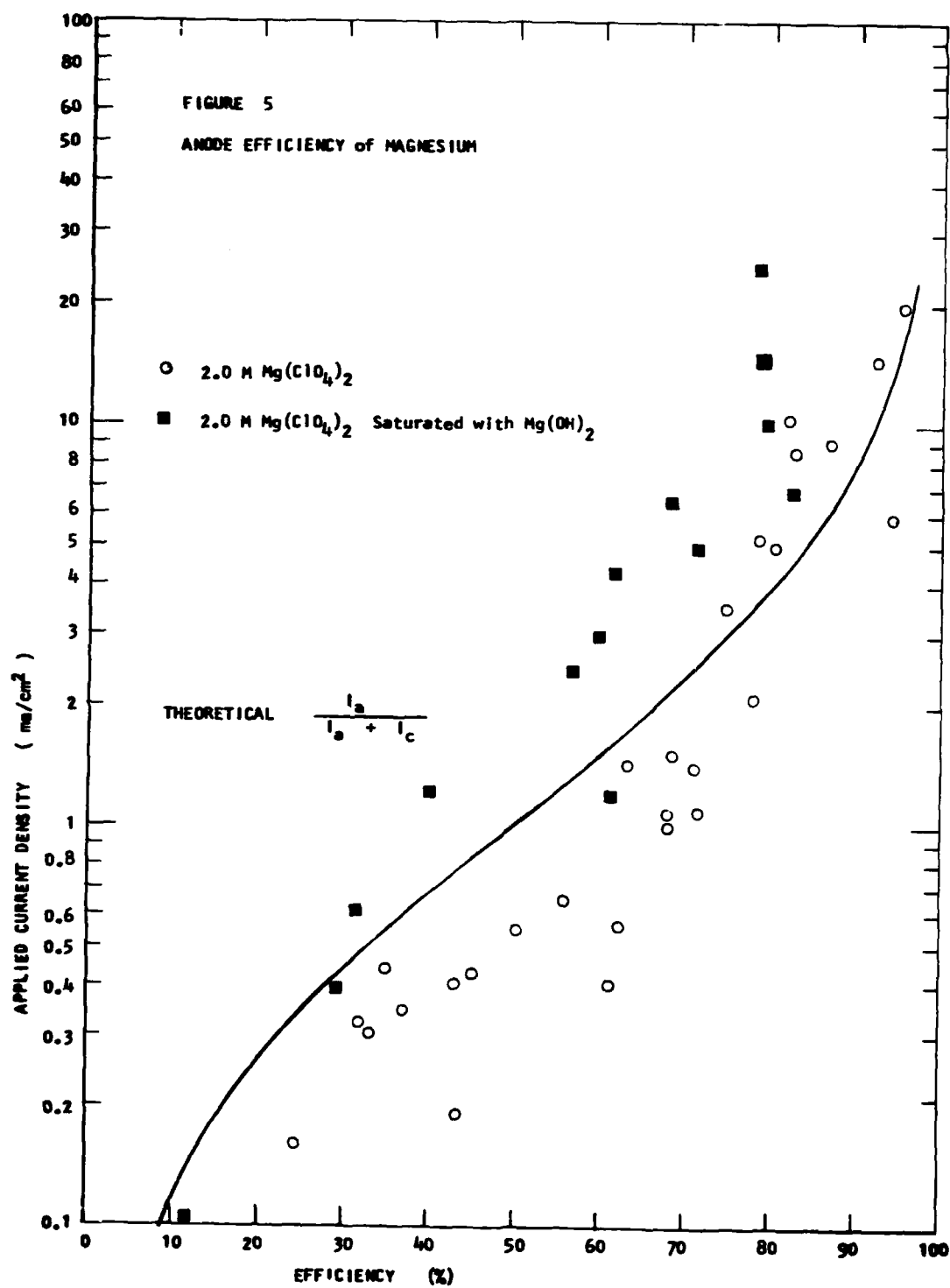
Results and Discussions of Data Concerning Efficiency

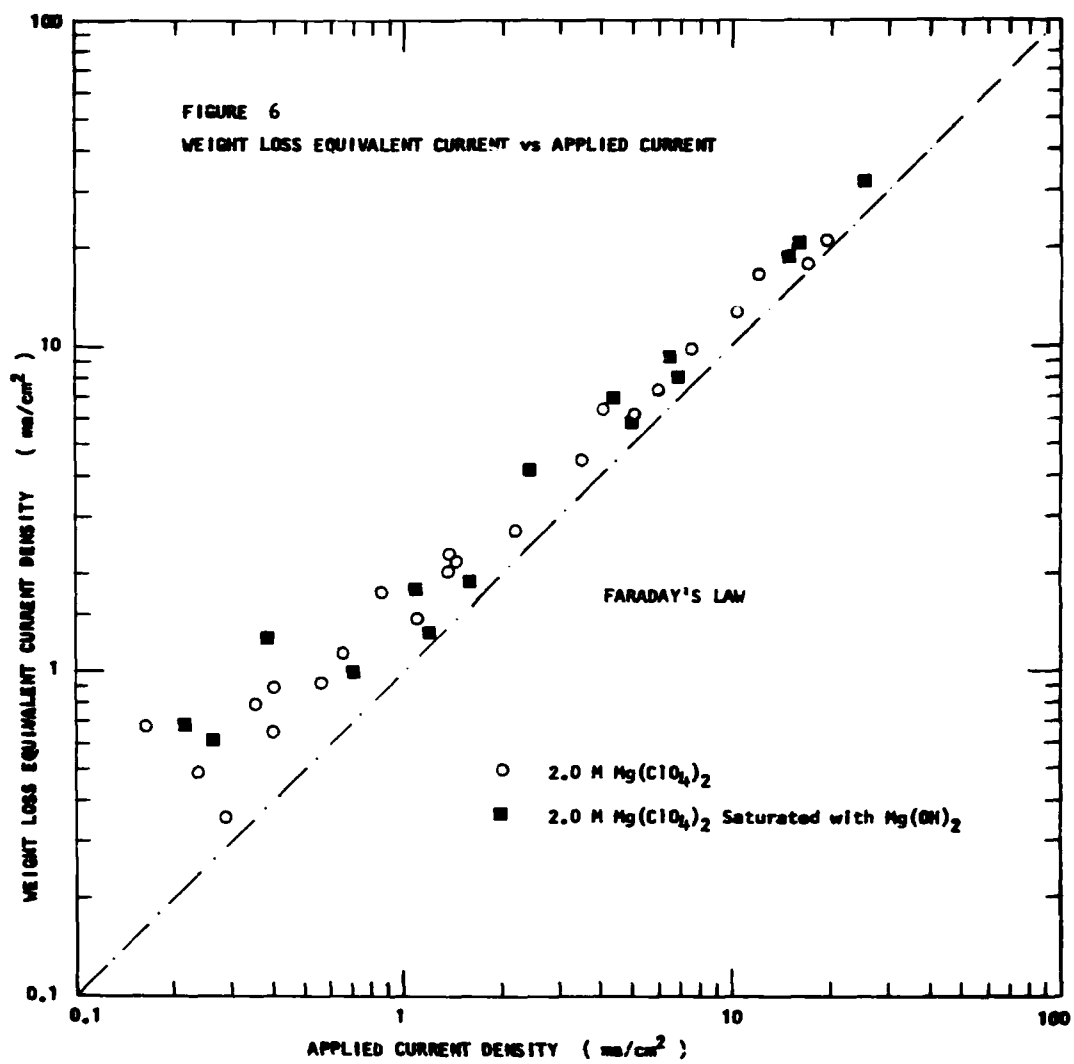
Electrolyte 1 - $\text{Mg}(\text{C}_2\text{O}_4)_2$ pH = 7.0

Observations and Discussions - When the Mg strips were removed from the electrolyte (see experimental set-up) they were found to possess 2 layers; the outer layer being a black-brown oily film, and the inner layer was gray-white in nature. The outer layer was

easily removed with a dry paper towel - while still in the electrolyte, it was found that the outer black brown layer would flake off if a sudden pulsating current was passed through the electrolyte. The inner gray white layer was removed as described previously. The small amount of localized pitting that was observed, occurred mostly on the fringes of the strip and at the air-electrolyte interface. The degree of pitting was related to the applied current - being minimal in the range of .1 to 5MA and slightly greater in the range of 5-30MA.

Figure 5 which shows anode efficiency as current density indicates that the efficiency of the $2M \text{ Mg}(\text{C}_2\text{O}_4)_2$ approaches the 90% region (94%) for a large current density (app. 20 MA/CM). This high calculated efficiency seems in part to negate the influence of the negative difference effect. This is further pointed out when we observe Figure 6 which is a measure of the applied current density vs the weight loss current density as per Faraday's Law. We see the curves to approach the values predicted by Faraday's Law for large current densities (app. 20 MA). This last point clearly refutes the results described by P.F. King⁽²⁾ (although his results are not for this particular electrolyte) in which it is shown that for large current densities there is a deviation from Faraday's Law. Figure 6 further shows the deviation to occur in the low current density range, whereas the results offered by P.F. King⁽²⁾ indicate a convergence in this region.



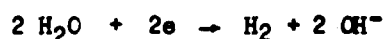
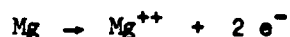


Electrolyte 2 - 2M $\text{Mg}(\text{C}_2\text{O}_4)_2$ Saturated with $\text{Mg}(\text{OH})_2$ pH = 7.0

Observations and Discussions - As in the case of the unsaturated $\text{Mg}(\text{C}_2\text{O}_4)_2$ (electrolyte 1), the Mg strips were found to contain the layers; the easily removed thin black-brown outer layer, and the gray white inner layer. Moderate localized pitting, surrounded by white precipitate, was observed at small current densities. It was further noticed that a sizeable amount of white precipitate was formed, accompanied by an increase in the pH reading. In addition no weight loss was observed for currents of 3 to 5 MA (the corresponding current density was 3 to 5 MA/cm^2) although for a current of 8 MA where current density was 4.23 MA/cm^2 a weight loss was observed.

The localized pitting that is observed, gives intuitive credence to the theory that suggests it is the impurities in Mg (AZ-21) that are responsible for this occurrence. As suggested by M.E. Straumanis and V.N. Wang⁽³⁾, an increase in corrosion of Mg with increased current density may be due to the destruction of anodic film (an hydroxide film) and the continuous exposure of impurity particles which serve as sites for further reactions to take place.

The white precipitate, believed to be $\text{Mg}(\text{OH})_2$, and the increased pH that was recorded during the run, indicate that the following electrochemical reactions are taking place:



On observing Figure 5 we note that the efficiency now approaches 78%. The magnesium anode in $\text{Mg}(\text{C}_2\text{O}_4)_2$ saturated with $\text{Mg}(\text{OH})_2$ electrolyte appears to be about 15% less efficient than in the unsaturated $\text{Mg}(\text{C}_2\text{O}_4)_2$. This relative poorer efficiency is however compensated for by the results shown in the Weight Loss Corrosion tests; namely, that the Mg strips is attacked to a lesser degree by the saturated electrolyte (35% weight loss for the unsaturated $\text{Mg}(\text{C}_2\text{O}_4)_2$ vs 17% weight loss for the same electrolyte, only now saturated with $\text{Mg}(\text{OH})_2$).

Figure 6 is again a refute of that predicted by P.F. King⁽²⁾ in that a convergence with Faraday's Law is observed at high current densities instead of the divergence predicted; and a divergence at low current densities where a convergence was expected.

Results and Discussions of Weight Loss Corrosion Tests

Table 3 shows the data for weight loss corrosion data in sealed bottles. It is observed that where phosphate ions are present (in relatively high concentration) the magnesium strips were not attacked to any great extent, notably a weight loss of approximately 0.5% for 1/9 electrolyte and approximately 5% for the $\text{Na}_2\text{HPO}_4/\text{Na}_2\text{CO}_3$ electrolyte.

An interesting comparison is made in observing the 2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ and the 2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ saturated with $\text{Mg}(\text{OH})_2$. When the $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ was saturated with $\text{Mg}(\text{OH})_2$, the percentage of weight loss was reduced from approximately 35% to approximately 17%. Experimental magnesium batteries now in use employ a $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ electrolyte saturated with $\text{Mg}(\text{OH})_2$. The above data points to the wisdom of saturating the electrolyte with $\text{Mg}(\text{OH})_2$, namely the increase of the battery's shelf life.

The indication of a white precipitate is attributable to the formulation of $\text{Mg}(\text{OH})_2$ according to the reaction.

The various colored layer formations that are visually observed, are believed to be the result of the reaction of the electrolyte with the various impurities found in the Mg (AZ-21); notably Al, Zn, Mn, Ni and Fe.

Table 3.

Weight loss corrosion tests - Sealed Bottles - Mg (AZ-21)

Duration = 320 hrs.

App. 50 ml. electrolyte/bottle

Electrolyte	Initial Wt.	Final Wt.	% Wt. Loss	Visual Observations
2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ pH = 7.0	1) .5643	.3480	38.4	Large amount of white
	2) .3630	.2568	28.2	ppt.= gray-gold speckled
	3) .3870	.2312	40.3	Layer= heavy pitting on edge
2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ Sat. w/ $\text{Mg}(\text{OH})_2$ pH = 7.1	1) .5646	.4781	15.3	Small amount of white
	2) .4692	.3900	16.9	ppt.= grey-gold layer
	3) .4111	.3346	18.6	Some spotty pitting on fringes

Conclusions

1. The corrosion rate of magnesium (AZ-21) in 4M $\text{NaC}^{\circ}\text{O}_4$ is about 1/100th the corrosion rate of magnesium (AZ-21) in 2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$.
2. Adding $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ to $\text{NaC}^{\circ}\text{O}_4$ increases the dissolution and corrosion of the magnesium.
3. The magnesium anode does not demonstrate the negative difference effect.
4. Saturating 2M $\text{Mg}(\text{C}^{\circ}\text{O}_4)_2$ with $\text{Mg}(\text{CH})_2$ decreases the corrosion rate of magnesium by about 50%.

Recommendations for Future Research

1. Current efficiency of Mg (AZ-21) in 4M NaClO₄, 3.96M NaClO₄ - 0.02M MgClO₄, and 3.6M NaClO₄ - 0.2M Mg(ClO₄)₂.
2. Corrosion Test on Mg (AZ-21) vs 4M NaClO₄, 3.96M NaClO₄ - 0.02M Mg(ClO₄)₂ and 3.6M NaClO₄ - 0.2M Mg(ClO₄)₂.
3. Fabricate a cell for test with above described electrolytes.
4. Study of reduction of m-DNB in 4M NaClO₄, 3.96M NaClO₄ - 0.02M Mg(ClO₄)₂ and 3.6M NaClO₄ - 0.2M Mg(ClO₄)₂.
5. Polarization curves of Mg dissolution in 3.6M NaClO₄ - 0.2M Mg(ClO₄)₂, 1.16M NaClO₄ - 0.02M Mg(ClO₄)₂ and 0.8M NaClO₄ - 0.2M Mg(ClO₄)₂.
6. Polarization curves for Mg dissolution in other cation in perchlorate electrolytes Li, Cs, Sr, Ba, Al, etc.

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- (1) L. Wikstrom, "Electrode-Electrolyte Interactions: Magnesium Anodes: meta-Dinitrobenzene Cathodes".
First Semi-Annual Report ECOM-0143-October 1968.
- (2) P. F. King, "The Role of the Anion in the Anodic Dissolution of Magnesium", J. Electrochem. Soc., 6, 536 (1966).

Identification of Key Personnel

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Akim Adubifa	Graduate Research Asst.	1/2 Time
Gerald Smaldone	Research Technician	1/3 Time
Bideon Stern	Research Technician	1/4 Time

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13. ABSTRACT The electrochemical dissolution of magnesium (AZ-21) and the h.e.r. on magnesium (AZ-21) in magnesium perchlorate, sodium perchlorate and a mixed electrolyte of sodium and magnesium perchlorate has been investigated. In the sodium perchlorate electrolyte, 0.4M to 4.0M, the Tafel Slope of the h.e.r. was unaffected by the direction of the potential sweep. In the magnesium perchlorate electrolyte, 0.2M to 2.0M, the Tafel Slope of the h.e.r. changes with direction of the potential sweep. The anodic Tafel Slopes in the pure electrolytes was 50 mv and 30 mv in the mixed electrolytes. The rate of magnesium dissolution in the magnesium perchlorate was about an order of magnitude faster than that in the sodium perchlorate electrolyte. On the other hand the corrosion current density of magnesium in the magnesium perchlorate was three (3) orders of magnitude greater than in the sodium perchlorate electrolyte. Corrosion rates of magnesium in magnesium perchlorate and magnesium perchlorate saturated with magnesium hydroxide was also investigated. It was found that addition of the hydroxide reduced the corrosion rate by about 50%. It was also determined that magnesium did not exhibit the Negative Difference Affect.		

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