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THE DOW CHEMICAL COMPANY REPORT

NINIH QUARTERLY REPORT
(DECEMBER 1, 1951 - FEBRUARY 29, 1952)
U. S. ATOMIC ENERGY COMMISSION
CONTRACT NO. AT(11-1)60

PREPARATION OF HIGH PURITY MAGNESIUM AND
A STUDY OF THE EFFECT OF NON-METALLIC AND
ALKALI METAL IMPURITIES ON THE CORROSION
CHARACTERISTICS OF PURE MAGNESIUM

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OF PURE MAGNESIUM

ABSTRACT:

Corrosion studies of the magnesium iron binary system indicate that the corrosion properties are significantly affected by the thermal history during casting. Three distinct types of thermal history are now recognized: 1. slow cooling ($\leq 2000^\circ\text{F}/\text{min.}$) from the pouring temperature; 2. rapid cooling (cold water quench $\sim 11000^\circ\text{F}/\text{min.}$) from the liquid state; 3. slow cooling ($\sim 100^\circ\text{F}/\text{min.}$) through the solidus temperature followed immediately by rapid quenching ($\sim 8000^\circ\text{F}/\text{min.}$ -cold water quench). The slow cooling results in a tolerance limit for iron in magnesium of ~ 60 ppm Fe. Alloys rapidly cooled from the liquid state have corrosion rates which are markedly improved over their slowly cooled counterparts. Nevertheless, they still show increases in corrosion rate above 60 ppm Fe. The third type of treatment has not been completely explored, but limited tests indicate that the corrosion rates for this treatment are relatively low and that a high tolerance limit similar to that of Hanawalt, Nelson, and Peloubet¹ may be observed. The solution potential measurements of Mg-Fe alloys also show a definite relationship to thermal history.

Corrosion evaluation of slowly cooled ($\leq 2000^\circ\text{F}/\text{min.}$) Mg-Fe-Si alloys revealed that 200 ppm silicon was significantly detrimental to the corrosion properties of pure magnesium containing 22 ppm Fe. Similarly, 2500 ppm silicon produced increased corrosion rates in the presence of 9 ppm Fe. On the other hand, silicon additions were beneficial in alloys containing 77 ppm Fe. Silicon had no effect on the solution potential of the lower iron content alloys, but did result in significant increases in the higher iron content alloys (77 ppm).

Investigations of the Mg-C and Mg-Fe-C systems revealed considerable segregation of the carbon as determined by chemical analyses. In spite of the high corrosion rates in certain cases, an evaluation of the data showed no definite evidence that carbon additions (10-140 ppm lampblack) are detrimental.

Re-evaluation of the corrosion properties of the Mg-Cl system refuted the conclusion drawn in the 4th quarter attributing increased corrosion rates in 3% NaCl to high chloride content.

Vacuum sublimation and argon casting was further investigated as a purification method for magnesium. It was not possible to correlate impurity content with corrosion rate of the high-purity metal. Certain physical factors not yet adequately defined may be masking the effect of impurity on the corrosion rate. Arc-melting of sublimed magnesium can be accomplished on a small scale, but the melt is slightly contaminated during the process.

INTRODUCTION:

The purpose of this investigation is to prepare high purity magnesium and to study the effect of non-metallic and alkali metal impurities on the corrosion characteristics of high purity magnesium. The first two years of the contract period were devoted primarily to a detailed study of purification by sublimation and to determine the effect of various non-metallic and alkali metal additions on the corrosion characteristics of pure binary magnesium alloys. During the 9th quarter the contamination studies were broadened to include the Mg-Fe system and ternary alloys of magnesium, iron and non-metallics. The pronounced effect of thermal history on the corrosion properties necessitated a study of this factor, particularly in the case of alloys containing minor amounts of iron (as low as 10 ppm).

SUMMARY OF EXPERIMENTAL RESULTS:

The corrosion properties of magnesium-iron alloys are remarkably sensitive to thermal history as a result of casting and heat treatment. Slowly (air) cooled alloys show increased

corrosion rates at iron contents as low as 30 ppm, although the tolerance limit is considered to be at 60 ppm. If the alloys are rapidly cooled by water quenching from the liquid state, vastly improved corrosion properties are obtained although somewhat increased corrosion rates prevail at and above approximately 60 ppm Fe (Fig. 1). This treatment also results in corrosion rates having a higher degree of scatter at the higher iron contents (140-310 ppm) than does slow cooling. By allowing the alloys to cool slowly ($\sim 100^\circ\text{F./min.}$) through the solidus temperature before quenching, the corrosion characteristics were improved to an even greater extent than by the quench from the liquid (Fig. 8). Thermal history affects not only the corrosion characteristics, but also the solution potential of the Mg-Fe alloys. For example, the quenched alloys undergo a 5mv decrease in potential upon increasing the iron content from 6 to 150 ppm while slow cooling results in an 50mv decrease with similar Fe contents.

Additions of silicon to Mg-Fe alloys may have a beneficial or detrimental effect upon the corrosion properties, depending upon the iron content of the alloys. Corrosion results indicate that silicon is injurious in alloys containing iron below the tolerance limit (Figs. 14, 15). Here silicon contents above 1000 ppm with 9 ppm iron or 150 ppm in the presence of 22 ppm iron result in increased corrosion rates. On the other hand, all silicon additions made to alloys containing 77 ppm iron (above tolerance limit) result in decreased corrosion rates (Fig. 16). In these higher iron alloys, increasing the silicon from 60 to 4800 ppm produced significant increases in the solution potential (1.648 to 1.660 mv). In contrast, silicon additions had no effect on the solution potential of lower iron content alloys (6 and 22 ppm).

The corrosion results of alloys containing 6, 20, and 66 ppm Fe with varying amounts of carbon (25-140 ppm lampblack), showed no high corrosion rates directly attributable to the carbon content. Similarly, the solution potential did not decrease as a result of increased carbon contents. Alloys contaminated with chlorides behaved in a similar manner to the carbon alloys since no harmful effect was detected in the 3% NaCl corrosion tests.

Phosphorus additions (12-4500 ppm) produced no change in the solution potential values of magnesium-phosphorus alloys in contrast to the sizeable effects previously noted in the Mg-Fe-P alloys. (Fig. 25, Biennial Report #COO-85).

Triple sublimation and argon casting of magnesium effectively reduces the iron content to 1 or 2 ppm as evaluated by chemical and neutron activation analyses, but fairly high carbon contents result. Corrosion rates of the sublimed and cast material appear to be influenced by certain physical factors such as cold working, thermal history, and surface roughness.

Arc-melting of triply-sublimed magnesium on a hearth of the same material was carried out in an attempt to produce a metal free from metallic impurity. The melt is contaminated with iron, manganese, and probably some oxide.

CONCLUSIONS:

Mg-Fe System:

It has been demonstrated that the cooling rates of the magnesium-iron alloys have a pronounced effect on their corrosion characteristics (Fig. 1). Furthermore, the tolerance limit for iron in magnesium as well as the solution potential values are changed by thermal history. A detailed metallographic study of the quenched alloys must be made before an explanation of these results is possible. Explanation of the vast differences in corrosion behavior between Mg-Fe alloys quenched from below and above the freezing point (Fig. 8) requires careful metallographic studies and possibly a determination of the phase diagram for this compositional region. The corrosion properties of Mg-Fe alloys quenched from temperatures below the freezing point should be investigated further.

Mg-Fe-Si System:

Silicon is detrimental to the corrosion characteristics of pure magnesium containing iron below the tolerance limit (~ 60 ppm), but salutary to alloys containing iron above the tolerance limit. No explanation for the deleterious effects of silicon in the presence of small amounts of iron is offered.

Comparison of the Mg-Fe-P alloys with the higher iron-silicon alloys suggests that the improved corrosion properties of the latter alloys may result from increased solubility of the cathodic second phase in the presence of silicon. The proposed metallographic examination of this system might clarify the role of silicon in the corrosion process.

Mg-Fe-C System:

Carbon (25-140 ppm) apparently had no harmful effect on the corrosion properties of high purity magnesium containing iron. This conclusion is proposed tentatively since only a limited amount of carbon (lampblack) could be introduced into the metal. Further exploration of this alloy system is believed necessary in view of the erratic corrosion results obtained on the SAC (Sublimation-Argon Cast) metal obtained in the purification program. Evaluation of the corrosion behavior of alloys containing ~ 40 ppm iron and varying carbon should reveal the effect of carbon on the tolerance limit for iron.

Mg-Cl System:-

Chlorides introduced into pure magnesium as #230 flux have no effect on the 3% NaCl corrosion properties provided the metal is free of other impurities.

Multiple sublimation and argon casting is effective in reducing the iron content of magnesium to 1 or 2 parts per million.

RESULTS AND DISCUSSION:

A. Contamination Studies:

1. Mg-Fe System:

Mg-Fe corrosion studies have been continued in an attempt to rationalize the variations in the Fe tolerance reported previously by the authors and that of Hanawalt, Nelson, and Peloubet¹. Earlier results of this project suggested a tolerance limit of ~ 60 ppm Fe, whereas Hanawalt et al reported 170 ppm. Thermal history appeared to be the most probable source of these tolerance limit variations since the casting techniques differed markedly. The first group of Mg-Fe alloys prepared for this

project were poured at a temperature of 800°C . into a split graphite mold, resulting in a cooling rate of approximately $2000^{\circ}\text{F./minute}$. Hanawalt, Nelson, and Peloubet allowed their alloys to cool slowly in the crucible through the solidus temperature; subsequently quenched them in a stream of running water, producing an estimated cooling rate of $\sim 10000^{\circ}\text{F./min}$. Greater solution of the cathodic second phase material (iron) would be expected from this type of treatment than from a slower cool. It is also conceivable that rapid cooling rates might result in better corrosion rates and account for a higher tolerance limit. The validity of the thermal history hypothesis was tested on a series of iron alloys quenched in still water from the melting point, giving a cooling rate of $11,000^{\circ}\text{F./min}$. The rapidity of the cooling rate during quenching should result in a super-saturated solid solution of Fe in Mg.

Corrosion tests of these alloys revealed that the alloys above 95 ppm Fe had two distinct zones which exhibited decidedly different corrosion rates. The exterior layer of the specimens which froze first was relatively resistant to corrosion in 3% NaCl while the interior of the specimens was seriously attacked. This behavior suggested that the interior of the specimens was liquid when quenched, whereas the exterior was solid before the quench. Additional melts purposely quenched from above and below the melting point substantiated this explanation. Quenching from the partially solidified state produced ingots with an exterior shell possessing high corrosion resistance and an interior which was rapidly attacked. On the other hand, alloys completely solidified before quenching showed no variation in the corrosion pattern, being generally quite corrosion resistant. Apparently, the cooling rate at or above the freezing point is as important as the cooling rate below the freezing point. The optimum conditions for enhanced corrosion resistance, appear to be a relatively slow cool through the freezing point followed immediately by a water quench.

Figure 1 illustrates the effect of thermal history on the corrosion characteristics of the Mg-Fe system. These data

show that the critical iron range (90-200 ppm) obtained on slower cooling ($\sim 2000^{\circ}\text{F./min.}$) is largely eliminated by rapid quenching ($11,000^{\circ}\text{F./min.}$) of the metal from the partially solidified condition. However, it was noted that the thermal history had no effect on the corrosion rates at the higher iron contents, 230-310 ppm, since the curves coincide in this range. The increased scatter in the experimental points obtained with the 3 hour alternate immersion exposure tests (3% NaCl) shown in Figure 2 for the quenched alloys may be attributed to the increased difficulty of obtaining reproducible rapid cooling rates. Higher Fe contents with similar thermal histories yield an apparently erratic corrosion behavior (cf. Fig. 2 and 3). However, additional work is necessary for clarification. Variations noted in the thermal history of alloys #70659 and #70656 have resulted in elimination of the corrosion data on these alloys from Figures 1, 2, and 3.

Although the present data has not revealed a tolerance limit for Fe as high as 170 ppm^1 consideration of the effect of thermal history on the corrosion characteristics would suggest that the tolerance limit may vary. It is anticipated that a series of Mg-Fe alloys cooled slowly through the freezing point and immediately quenched in cold water will furnish data closely paralleling the earlier results.

The corrosion characteristics obtained on slow air cooling (50°F./min.) or casting into a graphite mold ($2000^{\circ}\text{F./min.}$) are similar (See Fig. 1) in the iron range studied (35-100 ppm). In these cases increased corrosion rates are obtained if an iron content of $\approx 60 \text{ ppm}$ is attained. Similarly, if the iron content exceeds 60 ppm in alloys quenched from the partially solidified state, increased corrosion rates will also be obtained but the increases will be decidedly less than for the slowly cooled alloys.

The appearance of the corrosion specimens subsequent to 3 hour and 1 day alternate immersion exposures in 3% NaCl are shown in Figures 4, 5, and 6. Close examination of these figures will show a duplex structure exhibiting considerably different types of corrosion resistance in the higher iron content alloys.

Rapidly cooled and slowly cooled alloys containing 99 ppm Fe, illustrated in Figure 7, demonstrate the beneficial effect of

quenching the alloys. The variation in the corrosion properties of alloys containing 175 ppm Fe quenched from the solid and semi-solid state is shown in Figure 8. The improvement in corrosion resistance obtained by quenching after complete solidification is forcibly presented in this photograph.

If the tolerance limit of 170 ppm is assumed to be correct for rapid cooling, the tolerance limit for iron in magnesium must be controlled by the thermal history. Additional work will be necessary to prove this point conclusively.

Solution potential studies of the Mg-Fe alloys were conducted concurrently with the corrosion tests and will be discussed in a later section of the report.

2. Mg-C and Mg-Fe-C:

A series of Mg-C alloys were prepared by addition of equal parts of #230 flux (mixed chlorides) and lampblack to sublimed magnesium which contained approximately 9 ppm iron. Corrosion tests of these alloys gave erratic results which did not appear to be simply related to the carbon content. (Fig. 10). The considerable spread encountered with specimens having duplicate carbon analyses suggests that the erratic corrosion results may be attributed to segregation of the carbon additions which possess limited solubility in the magnesium. Detailed analyses of these alloys revealed that two compositions - (#70398 and #70402) - contained rather large amounts of chlorides, 76-483 and 19- 140 ppm Cl respectively. This difference in chloride content might be causing the erratic corrosion behavior. However, subsequent work demonstrated that chloride additions alone have little effect on the salt water corrosion characteristics of high purity magnesium, (Fig. 21)(See section on chloride contamination). However, chlorides cannot be disregarded completely as a source of erratic corrosion behavior since combinations of carbon and chlorides or iron, carbon and chlorides may be detrimental to the corrosion properties of magnesium. More complex combinations of these constituents will be required to define their relationship.

In the subsequent alloy series of Mg-Fe-C (#70385-70388, 70390-70394) attempts were made to prepare melts containing 20 and

40 ppm Fe with varying amounts of carbon (lampblack). Analysis of these alloys revealed average iron contents of 20 and 66 ppm and average chloride contents of 7 and 10 ppm -- the maximum reported chloride was 21 ppm. The widespread variations encountered suggested that segregation was present in the alloys.

The corrosion behavior of the lower iron alloys (Fig. 11) indicate that 130 ppm carbon may be a slightly injurious to the corrosion resistance of high purity magnesium containing 20 ppm iron.

Since iron contents on the order of 60 ppm (tolerance limit for cooling rate of 2000°F./min.) give disproportionately large corrosion rates, it is unfortunate that attempts to make alloys containing 40 ppm Fe yielded an average iron content of 66 ppm Fe. Consequently, the task of separating the combined effects of carbon and iron on the corrosion rate became more difficult. However, consideration of the data presented in Figure 12 leads to the conclusion that the corrosion values are readily explainable on the basis of the iron content. Apparently, 100 ppm carbon have no detectable effect on the salt water corrosion resistance of pure magnesium containing 66 ppm iron. A series of Mg-Fe-C alloys containing 40 ppm Fe will be necessary to decide whether the tolerance limit for iron in magnesium can be lowered by the presence of suitable amounts of carbon. Further work will include this series of tests.

3. Mg-Si and Mg-Fe-Si:

Powdered silicon additions (3-660 ppm) were made to melts (#68270-68276) of sublimed magnesium (4-5 ppm Fe) for the purpose of determining its effect on the corrosion properties. The corrosion data for these alloys (Fig. 14) indicate that silicon above 1000 ppm increased the corrosion rates. Because the alloys appear to pick up approximately 3 ppm Fe/1000 ppm Si, some ambiguity exists as to whether the increased corrosion rates result from a lowered iron tolerance limit or from the detrimental influence of silicon in iron-free Mg. If the former were true, a tolerance limit of 10 ppm Fe would be indicated in the presence of 2500 ppm silicon. However, justification for this argument may

be found in Figure 15, which illustrates the effect of silicon on the corrosion properties of magnesium containing 22 ppm Fe. Here it will be noted that the tolerance limit for silicon is only 150 ppm in the presence of 22 ppm iron when the alloys are relatively slow cooled ($\sim 2000^{\circ}\text{F./min.}$).

When the iron content is above the tolerance limit (~ 60 ppm when cooled at $= 2000^{\circ}\text{F./min.}$), the effect of silicon additions appears to be entirely beneficial, (Fig. 16). As this shows, the beneficial effects on the corrosion characteristics are nearly proportional to the magnitude of the silicon additions (60-2300 ppm). Since these alloys exhibit decreasing corrosion rates at 2300 ppm silicon, the work will be extended to higher concentrations of silicon with the same iron content (77 ppm). Studies of the corrosion characteristics of the pure magnesium-silicon binary system will be continued. A determination of the silicon tolerance limit in the presence of 40-50 ppm Fe should also be informative. It is believed that the beneficial effects of silicon in the higher iron alloys may be a result of an increased solubility of the iron analogous to the previously reported effect of phosphorus. Microstructural studies of these alloys will be undertaken to confirm this idea.

4. Chloride Contamination:

Determination of the effect of chloride contamination on the corrosion behavior of sublimed magnesium was accomplished by additions of #230 flux (Melts #68409-68415). Alloys containing 40 to 2500 ppm of chlorides with an average iron content of 7 ppm were produced by casting the alloys without the usual settling period. The corrosion results and the scatter in analytical results shown in Figure 21 indicate that chlorides as high as 2500 ppm have no detrimental effect on the 3% NaCl corrosion resistance even in the presence of 7 ppm Fe. These results show further that the high corrosion rates attributed to chloride content in the 4th quarter (#COO-21, Fig. 5 & 6) should probably be attributed to Fe + Cl in conjunction with a relatively slow cool. A study of the Mg-Fe-Cl system is planned to determine whether or not chlorides plus minor amounts of iron (20-40 ppm) have a detrimentally additive effect on the corrosion properties of high purity magnesium.

5. Solution Potential Measurements:

Solution potential measurements were made on the alloys prepared in recent quarters in order to determine what element and/or combination of elements produce alloys which are cathodic to high purity magnesium. It was thought that this data might aid in deciding which of those elements have a deleterious effect on the corrosion characteristics of high purity magnesium.

The non-metallics; silicon, carbon and phosphorus show no effect on the solution potential of pure magnesium (Figs. 13, 17, and 20). However, when a cathodic impurity such as iron is present, the effect of such elements can be quite marked. In the Mg-Fe-P alloys it has been reported (Biennial Report, #COO-85) that phosphorus can raise or lower the solution potential values depending upon the iron content and the amount of phosphorus present. Below the iron tolerance limit (~ 60 ppm) phosphorus effects are negligible; above this iron value the solution potential is decreased by small amounts of phosphorus and increased by large amounts of phosphorus. Because these effects vary with iron content, the reader is referred to Figure 22 (Biennial Report, #COO-85) for information on specific compositions.

Silicon affects the solution potential of Mg-Fe alloys in a similar manner to phosphorus in spite of its different corrosion behavior in alloys containing iron below the tolerance limit. Silicon did not change the solution potential of alloys containing 22 ppm Fe (Fig. 18), but did increase the solution potential of alloys containing 77 ppm Fe (Fig. 19).

No effect of carbon on the solution potential values of Mg-Fe alloys could be detected (Fig. 13) because of the limited range of carbon additions. Different results, however, might be expected if a method of adding greater amounts of carbon could be devised.

The solution potential values of Mg-Fe alloys rapidly quenched (11,000°F./min.) from the partially solidified condition are shown in Figure 9. These data show a slight decrease (8 mv) in solution potential from 6-310 ppm Fe. This curve differs markedly from that obtained on a series of slow cooled (2000°F./min.)

Mg-Fe alloys (Fig. 22, Report #COO-85). The implications of this pronounced difference in solution potentials are not clearly understood at this time since a metallographic study of the new alloys has not been completed. No differences in solution potentials between the interior and exterior of the specimens were detected in spite of the sizeable differences in corrosion behavior.

More precise data on the relationship between the solution potential and the iron content (slow cooled alloys) would undoubtedly aid in a better understanding of the Mg-Fe ternary corrosion data. Data on this relationship are currently being obtained in conjunction with corrosion data on alloys containing 40-100 ppm Fe.

6. Effect of Thermal History on Iron Analyses:

The non-reproducibility of iron analyses encountered in the earlier work resulted in considerable conjecture as to the cause of these discrepancies. Consequently, the effect of extruding and casting Mg-Fe alloys at various temperatures was investigated.

a. Extrusion:

No significant effect on the spectrographic analyses for iron was discovered by extruding two alloys containing different concentrations of iron at low or high temperature. See table below:

Alloy No.	Description	Spec. Fe Anal. (ppm)
69320	"As Cast"	330
69320-1	"As Extruded" 900°F. (nose)	330
" -1	" " " (butt)	"
69320-2	" " 650°F. (nose)	335
" -2	" " " (butt)	330
69324	"As Cast"	188
69324-1	"As Extruded" 900°F. (nose)	188
" -1	" " " (butt)	190
69324-2	" " 650°F. (nose)	180
" -2	" " " (butt)	200

b. Casting:

An alloy (#67515) containing approximately 310 ppm Fe was sampled with a pneumatic gun at 50°F. intervals from 1200 to 1400°F. The duplicate spectrographic analyses are plotted against increasing and decreasing temperature in Figure 22.

Casting Temperature	<u>1200</u>	<u>1250</u>	<u>1300</u>	<u>1350</u>	<u>1400°F.</u>
Iron Content (ppm)	320	315	305	335	315
(Increasing Temp.)	310	270	270	300	310
Iron Content (ppm)	275	315	360	375	
(Decreasing Temp.)	265	285	330	330	
Average Fe (ppm)	293	296	316	335	313
Average Standard Deviation (σ) ppm Fe	23	19	33	70	3

The average analysis and average standard deviation calculated for each casting temperature are given in the above table. Comparison of these analytical results with those of the extruded alloys (preceding table), reveals a greater degree of scatter in the cast alloys. Since the analyses of the extruded alloys demonstrate excellent precision for the analytical method the high degree of analytical scatter for the cast metal suggests an effect of casting temperature. Although no direct relationship between casting temperatures and spectrographic analysis can be seen at this time, a trend toward higher iron values is noted up to 1350°F. The considerably higher average standard deviation values at 1300 and 1350°F. as compared to 1400°F. warrant further investigation.

B. Purification Studies:

1. Vacuum Sublimation-Argon Casting Unit:

a. Summary of Operations:

The purification program using the previously described subliming and casting unit (see Biennial Report #COO-85, this project, 1951) was continued during this quarter. A series of three sublimations and castings was completed using a graphite charge cradle and graphite entrainment barrier. The poor heat transfer of the graphite linings resulted in low yields (Table III), but 8 lbs. of triply sublimed and cast magnesium was produced.

b. Analysis of Purified Magnesium:

Spectrographic, chemical, and neutron activation analyses of the metal produced in this series are listed in Table I under alloy numbers 67536, 67537, and 67538 (SVC^{**} runs 13, 14, and 15). The largest single impurity present is carbon, as shown by chemical analysis. In 67538, 0.0107% of carbon was reported, probably introduced primarily by the acetylene black coating given the graphite lining to prevent sticking of the cast billet. This same lampblack treatment need not introduce more than 14 ppm of carbon to the cast billet, however, when the billet is large enough to increase the ratio of Mg:C as in alloy no. 67536.

The erratic nature of the carbon analyses indicates that carbon is somewhat segregated in the purified metal. Preliminary metallographic studies of 67538 bear out this observation.

Neutron activation analyses for iron in alloy numbers 67532, 67534, and 67535 were completed by the Spectroscopy Laboratory and are listed in the following table together with chemical and spectroscopic values. Neutron activation appears to be an excellent method for analyzing magnesium having extremely low iron contents. An ultimate sensitivity of a few hundredths of a part per million is indicated:

Alloy No.	No. of Subl.Rec'd.	Spect.	Chemical Fe ppm		Neutron Activation
			Acid Wash ¹	Machined ²	
67532 B	1	18	12.6		7.08±1.4%
T		20	"		"
67534	2	4	1.0		0.90±1.9%
67535 A	3	9	1.7		1.43±1.7%
B		11	"		"
C		4	"		"
D		6	"		"
E		4	"		"
F		4	"		"
G		4	"		"
H		4	"		"
I		11	"		"
67536	1	5	3	7	
67537	2	7	1.2	2.4	
67538	3	6	1.6	2.6	

1. Samples parted with hacksaw and acid washed.

2. " " " " "

^{**} Abbreviation for "Sublimation-Vacuum Casting"

An examination of the above analyses shows that in every case, the spectrographic analyses are considerably higher than the chemical and neutron activation analyses. Since spectrographic results are standardized initially against chemical analyses, it would seem advisable to accept the chemical results as being more nearly correct. In the instances where both the chemical and neutron activation analyses are available, it is considered that an average of the two results is the best value. The agreement between these latter two methods is seen to be quite good, particularly at the low values.

With reference to the preceding table, it is concluded that the iron impurity in magnesium can be reduced to 1 part per million by the process of multiple sublimation and casting. It is interesting to observe that no further purification with respect to iron is obtained after the second sublimation and casting. It may be that iron leached from the graphite lining (which itself contains .3-.5% iron) introduces this degree of contamination.

The Spectroscopy Laboratory is cooperating in trying to determine the cause of the discrepancy between the methods of analysis. The results of their investigation will be reported at a later date.

c. Cooling Rate of SVC Metal:

Because of the known effect of thermal history on the corrosion rate of magnesium and its alloys, a typical cooling curve of the sublimed and cast billets (SVC-7 through SVC-15) is included (Fig. 24). It is seen that the well insulated vacuum furnace results in an extremely slow cooling rate.

d. Observed Corrosion Rates of High-Purity Magnesium:

The corrosion rates of various sublimed and cast magnesium samples are given in the following table together with the concentrations of critical impurities¹:

¹ See also page 14 for discussion of Fe analyses.

Alloy No.	Subl. Rec'd.	Analyses ppm			Corrosion Rate, mcd	Exposure Time (days)
		Fe	Mn ^(a)	C ^(c)		
67530	2	5	(a)	2	.26	14
"	"	"	"	"	.28	14
67531	4	11	(a)	"	.33	7
67532	1	9.8	(b)	18	.87	14
67533	4	5	(a)	2	.34	14
"	"	"	"	"	.20	14
67534	2	.95	(b)	2	.32	14
67535	3	1.6	(b)	1	.32	14
67536	1	3	(c)	7	.34	14
67537	2	1.2	(c)	3	80 ± 40 .28	14

(a) Spectrographic results

(b) Average of neutron activation and chemical results

(c) Chemical results

* Alternate immersion, 3% NaCl, average of 3 samples.

It can be seen that there is no apparent correlation between the amount of impurity and the observed corrosion rate (with the exception of "high" iron in 67532). However, due to possible segregation of impurities in the cast billet, analyses might not represent impurity content in corrosion specimens. Therefore, without care in sampling, correlation of analyzed impurity content with corrosion rate might lead to erroneous conclusions. Certain SVC billets (67532, 67533, and 67535) were rather thoroughly analyzed, and the results show that this possibility cannot be overlooked (see Table I, Biennial Report #COO-85, this project).

Examination of the preceding table also shows that the more highly purified samples have no better corrosion rates, generally, than singly-sublimed material which has been melted with flux in graphite crucibles. It appears that there are several physical factors heretofore overlooked which may be affecting the corrosion rate of magnesium, but which are masked in materials having reasonably high corrosion rates. As the purity of the metal is increased to where the effect of cathodic impurity on the corrosion rate is slight, it is conceivable that these other effects may

become noticeable. Several such factors are listed below together with a brief discussion of their possible influence on corrosion rate:

(1) Varying thermal history:

It has been shown that the corrosion rate is influenced by the thermal history of the material. This is primarily due to the precipitation or solution of a cathodic second phase, however, and as such, is directly related to the impurity content. There is the possibility that even in high-purity magnesium, varying thermal treatments may alter the corrosion rate. Rapid cooling might introduce residual internal stresses giving rise to stress induced corrosion. Annealing would be expected to relieve any such residual stresses and result in lower corrosion rates.

(2) Varying amounts of cold working:

In the preparation of corrosion specimens (machining, grinding, etc.), the specimen is subjected to a certain amount of cold working. The resultant surface stresses could conceivably affect the corrosion rate adversely. This, indeed, is indicated by an examination of corrosion patterns on high-purity magnesium. There is a definite tendency for corrosion to follow the direction of machining on the specimens.

(3) Surface contamination:

During specimen preparation, surface impurities may be introduced in amounts sufficient to initiate electrochemical attack.

(4) Surface roughness:

Due to the phenomenon of crevice corrosion which magnesium exhibits, surface roughness may play an important role in the corrosion rate of high-purity magnesium when the specimens are prepared by certain methods such as grinding or machining. Also, since corrosion rates are given in weight loss per unit area per unit time, it is important that the surface area be correctly known. It is reasonable to assume that if we consider two specimens of the same exterior dimensions, but having varying smoothness, the rougher specimen will have a larger actual

surface area by virtue of its roughness. This should be considered in comparing corrosion rates of high-purity samples having varying surface preparations.

(5) Varying solution potentials and hydrogen overvoltages in different crystallographic directions:

Even if other effects could be completely eliminated, one must consider the possibility that the solution potential and hydrogen overvoltage might be different for one crystal face than for another. This could allow a local cell type of corrosion to occur in the absence of impurity.

It seems likely that all of these factors must be considered in the salt water corrosion of high-purity magnesium. At this point in the program, it would seem advisable to investigate the relative magnitudes of the above factors in order to determine the necessity for further purification studies. We may have reached a degree of purity where further removal of impurities has such a slight effect on corrosion rate as to be negligible in comparison with these other factors.

One interesting observation is that Alloy No. 67532, with approximately 10 ppm of iron, has the fairly high corrosion rate of 0.9 mcd. This is quite evidently caused by impurity since the specimens pit appreciably on 14 day test. It is concluded that slow cooling (Fig. 24) can cause as little as 10 ppm of iron to affect the corrosion rate of magnesium. This indicates an extremely low solid solubility of iron in magnesium at low aging temperatures.

2. Tantalum-Lined Purification Unit:

In order to eliminate the last major source of contamination, namely carbon, the feasibility of operating a small, tantalum-lined sublimation and casting unit is being considered. Tantalum is known to have a very low solubility in molten magnesium^{*} and should be an excellent liner material for a purification unit. Two disadvantages in constructing such a unit would be the high cost involved, and the fact that magnesium bonds to tantalum making removal of the solidified magnesium from the unit

^{*} Met. Lab. Report No. 11699, File No. 151

difficult. These disadvantages might be overcome by lining the casting crucible with a thin tantalum foil inexpensive enough to be thrown away. The foil could be machined off of the purified magnesium after removing the solidified melt from the unit.

3. Arc-Melting of Sublimed Magnesium:

In order to provide a means of remelting high-purity sublimed magnesium without crucible contamination, as-sublimed magnesium was arc-melted using the pure metal for both the electrode and hearth.

a. Apparatus and Procedure:

A diagram of the melting pot used for this series of experiments is given in Figure 23. A hearth of triply sublimed magnesium (69629-1) was sawed out of as-sublimed material and placed in the grounded melting pot. An electrode (approximately $3/4"$ x $3/4"$ x $12"$) was broken from the as-sublimed metal with as little handling as possible. Argon gas was introduced at an appreciable rate through the two small inlet tubes at the bottom of the melting pot to provide a protective atmosphere for melting. Then, using a standard Lincoln arc-welding machine, a puddle of magnesium 1 to 2 inches in diameter and $1/2$ inches thick was arc-melted on the hearth. Clouding of the sight glass by vaporized magnesium was prevented by the argon entering adjacent to the sight glass.

One problem encountered in the above procedure was the difficulty in maintaining an arc, because the electrode melted and dropped off in large drops. With currents up to 300 amperes and using reverse polarity, however, it was possible with practice to obtain melts of the above-mentioned size.

b. Analysis Results:

Of the metallic impurities spectrographically analyzed for (69629-2, Table I), only iron and manganese were detected. In view of the discrepancies discovered between the spectrographic, neutron activation, and chemical analyses of SVC magnesium (see page 14), the analysis results for 69629-2 may be somewhat high. The amount of iron present will be checked chemically to see if there is actually 12 ppm of iron present. It is entirely possible, however, that the observed impurities were introduced during the handling prior to arc-melting.

c. Metallographic Examination:

A metallographic examination was made of the triply sublimed and arc-melted magnesium (Alloy Nos. 69629-2, -3, -4). Figure 25 shows that the metal is quite clean except for a few rather irregular inclusions. These are probably magnesium oxide particles formed when the sublimed crystals are exposed to the atmosphere before the arc-melting step.

d. Corrosion Results:

Fourteen-day corrosion rates were obtained on the arc-melted metal with the following results:

<u>Alloy No.</u>	<u>Analysis Results ppm</u>		<u>Corrosion Rate (mcd)</u>
	<u>Fe</u>	<u>Mn</u>	
69629-1	2	2	---
69629-2-1	12	2	0.31
69629-2-2	---	-	0.34
69629-3	---	-	0.37
69629-4*	---	-	0.50

* Undersized sample

It is seen that the observed corrosion rates are no lower than those of ordinary singly-sublimed magnesium remelted with flux in graphite. This may be attributed to the apparent contamination by the iron. If it is shown chemically that iron isn't present in these amounts, however, the significance of the discussion on the corrosion of SVC magnesium may well apply here (page 17).

REFERENCE:

- ¹ Hanawalt, Nelson, and Peloubet, T.P. No. 1353-E 353, A.I.M.E., September 1941.

TABLE I

NUMERICAL LISTING OF MAGNESIUM ALLOY ANALYSES (In Parts Per Million)

AVERAGE CORROSION DATA - ALTERNATE IMMERSION IN 3% NaCl

AVERAGE CORROSION DATA - ALTERNATE IMMERSION IN 3% NaCl																									Average	Exposure		
Alloy No.	Subl. Run No.	Subl. Rec'd.	Description	Al	Cu	Fe ¹	Mn	Ni	Pb	Si	Sn	Zn	Zr	Ba	Ca	K	Na	Sr	C	H	P	B	Cl	M	Miscellaneous	Corrosion Rate	Time (Days)	
Sublimed Magnesium																												
67530	SVC-7	2	Sublimed, As Cast Mg.	1	1	5	<2	<3	<5	<10	<10	<100		<1	13	7	10	<1	29	20							.26	14
67531	SVC-8	4	"	<1	8	11	"	"	7	"	"	"		2	15	8	10	"	24							Li = <25	.33	7
67532	SVC-9	1	"	3	5	9.8 ^a	18	"	6	"	"	"		33	10	5	19	"	14	2			5			B = <1	.87	14
67533	SVC-10	4	"	<1	<1	5	2	"	5	"	"	"		<1	10	19	19	"	36	4						B = <1	.34-.20	14
67534	SVC-11	2	"	2	"	9.5 ^a	<2	"	6	"	"	"		"	"	13	10	"	38								.32	14
67535	SVC-12	3	"	<1	"	1.6 ^a	<1	"	<5	"	"	"		"	15	6	13	"	20								.32	14
67536	SVC-13	1	"	<300	"	3 ^a	7	"	"	"	"	"		"	<5	11	15	"	14	4						Cr = <1	.34	14
67537	SVC-14	2	"	--	8	1.2 ^a	3	"	"	"	"	"		"	"	11	11	"	80±40	5			3				.28	14
67538	SVC-15	3	"	"	9	1.6 ^a	2	"	"	"	"	"		"	"	10	9	"	107	14			2					
68859	S-61	1	Sublimed Mg	2	<1	2	<2	<3	<5	<10	<10	<100		<1	10	11	27	<1										
59629-1	S-68	3	Sublimed Mg	<300	"	2	"	"	"	"	"	"																
59629-2	S-68	3	Subl., arc-melted Mg	"	"	12	2	"	"	"	"	"														.34	14	
59530	S-69	1	Sublimed Mg	"																								
Mg-Fe Alloys																												
70655	#67515 only (Mg - 370 ppm Fe)			3	285	8	<3	10	<10	<10	<100			<100												14.27	1/8	
70656	#67515 + #68859 (sublimed Mg)			1	265	7	<3	7	"	"	"			"												104.1	1	
70657	#67515 + #68859			2	205	8	"	8	"	"	"			"												4.52	1/8	
70658	"			1	244	7	"	7	"	"	"			"												24.0	1	
70659	"			<1	170	6	"	6	"	"	"			"												14.58	1/8	
70660	"			"	185	5	"	<5	"	"	"			"												58.7	1	
70661	"			"	132	5	"	"	"	"	"			"												18.92	1/8	
70662	"			1	170	6	"	6	"	"	"			"												19.7	1	
70663	"			1	138	5	"	"	"	"	"			"												3.94	1/8	
70664	"			1	120	5	"	"	"	"	"			"												1.08	1	
70665	"			1	134	5	"	"	"	"	"			"												7.54	1/8	
70666	"			1	98	5	"	"	"	"	"			"												10.4	1	
70667	"			1	100	5	"	"	"	"	"			"												8.14	1/8	
70668	"			1	83	5	"	"	"	"	"			"												7.91	1	
70669	"			1	75	5	"	"	"	"	"			"												4.93	1/8	
70670	"			1	57	4	"	"	"	"	"			"												2.16	1	
70671	"			1	47	4	"	"	"	"	"			"												4.07	1/8	
70672	"			1	28	4	"	"	"	"	"			"												1.47	1	
70673	"			1	9	4	"	"	"	"	"			"												3.38	1/8	
70674	"			1	9	4	"	"	"	"	"			"												1.14	1	
70675	"			1	9	4	"	"	"	"	"			"												2.54	1/8	
70676	"			1	9	4	"	"	"	"	"			"												1.09	1	
70677	"			1	9	4	"	"	"	"	"			"												2.97	1/8	
70678	"			1	9	4	"	"	"	"	"			"												.635	1	
70679	"			1	9	4	"	"	"	"	"			"												2.77	1/8	
70680	"			1	9	4	"	"	"	"	"			"												.521	1	

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TABLE I (3)

Alloy No.	Description	Al	Cu	Fe	Mn	Ni	Pb	Si	Sn	Zn	Zr	Ba	Ca	K	Na	Sr	C	H	P	S	Cl	N	Miscellaneous	Average Corrosion Rate (mcd)**	Exposure Time (Days)
<u>Pure Mg-C Series</u>																									
70398	#68859 only (blank)		2	7 7	3	<3	<5	<10	<10	<100							59-148					76-483		1.7-145 4.6-10.2	1/8 7
70399	#68859 + 3 gms lampblack		1	7 10	3	"	"	"	"	"							21-25					6		.96 .384	1/8 7
70400	" + #70399		2	12 18	4	"	"	"	"	"							16-39					12		1.17 .393	1/8 7
70401	" + #70400		4	14 8	3	"	"	"	"	"							13-37					14		.652 .404	1/8 7
70402	" + #70401		<1	5 6	2	"	"	"	"	"							<10-50					19->140		.5-1.2 .358	1/8 7
				9 Ave.																					
<u>Mg-Fe-Si Series</u>																									
70433	#70383 only (blank)		2	23 22	9	<3	<5	3* 3	<10	<100														1.59 .49	1/8 1
70434	#70383 + 0.7 gms Si		1	25 22	9	"	"	1490* 1900*	"	"														2.97 40.0	1/8 1
70435	" + #70434		1	19 20	8	"	"	490* 370*	"	"														1.56 2.14	1/8 1
70436	" + #70435		2	19 22	8	"	"	350* 210*	"	"														2.21 1.88	1/8 1
70437	" + #70436		1	26 22	9	"	"	170* 140*	"	"														1.65 .498	1/8 1
				22 Ave.																					
70438	#70384 only (blank)		1	70 76	13	<3	<5	60* 60	"	"														162 Destroyed	1/8 1
70439	#70384 + 0.7 gms Si		2	77 83	14	"	"	1480* 2300*	"	"														24.4 172	1/8 1
70440	" + #70439		3	74 79	13	"	"	540* 560*	"	"														85 114	1/8 1
70441	" + #70440		2	77 80	13	"	"	130* 130*	"	"														125 Destroyed	1/8 1
70442	" + #70441		1	74 76	13	"	"	90* 90	"	"														137 Destroyed	1/8 1
				77 Ave.																					
<u>Mg-Fe Master Alloys</u>																									
70468	#68859 + #67515																								
70469	"																								
70470	"																								
70471	"																								
70472	"																								

- 1 Unless Otherwise Noted, results are spectrographic
 2 Average of neutron activation and chemical analyses.
 3 Chemical analysis.
 4 Single test.

- * Chemical Analyses
 ** See Table II for details

TABLE II

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DETAILED CORROSION AND SOLUTION POTENTIAL DATA

Alloy No.	Composition (ppm)	Melt Size	Thermal History	Alternate Immersion Corrosion Rate (mcd) [*]			Solution Potential (Volts)
				3 hour	Exposure 7 day	14 day	
67530	Sublimed and cast Mg	25 lbs.	Slow cool (Fig 24)			.26	
67531	(See Analysis Results)	25 "	(")		.33		
67532	(")	85 "	(")			.87	
67533	(")	24 "	(")			.34 - .20	
67534	(")	58 "	(")			.32	
67535	(")	29 "	(")			.32	
67536	(")	49 "	(")			.34	
67537	(")	25.5 "	(")			.28	
67929-2,3	3X sublimed and arc-melted Mg (Fe = 12 ppm)	5-10 gms.	Shock cooled			.34	
[*] Triplicate determinations							
<u>Mg-Fe-C Series</u>							
70385-1	17-19 Fe	131 - 149 C	135 gms.	800°C.	1.56		1.667
-2	18 Ave.	140 Ave.	135 "	"	1.87		1.667
-3			135 "	"	1.71 Ave.	.390	1.665
-4			135 "	"		.366	1.666 Ave.
						.378 Ave.	
70386-1	20-23 Fe	98 - 116 C	135 "	800°C.	.993		1.667
-2	21 Ave.	107 Ave.	135 "	"	.998		1.666
-3			135 "	"	.995 Ave.	.403	1.672
-4			135 "	"		.367	1.668 Ave.
						.385 Ave.	
70387-1	18-24 Fe	<10 - 212 C	135 "	800°C.	1.37		1.668
-2	21 Ave.	106 Ave.	135 "	"	.685		1.668
-3			135 "	"	1.03 Ave.	.332	1.666
-4			135 "	"		.400	1.667 Ave.
						.366 Ave.	
70388-1	18-22 Fe	29-39 C	135 "	800°C.	1.06		1.666
-2	20 Ave.	34 Ave.	135 "	"	.999		1.667
-3			135 "	"	1.03 Ave.	.335	1.665
-4			135 "	"		.288	1.666 Ave.
						.312	
70390-1	62-66 Fe	32-88 C	135 "	800°C.	.686		1.661
-2	64 Ave.	60 Ave.	135 "	"	1.25		1.661
-3			135 "	"	.868 Ave.	.483	1.661
-4			135 "	"		.472	1.661 Ave.
						.477	

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TABLE II (2)

Alloy No.	Composition (ppm)		Melt Size	Thermal History	Alternate Immersion Corrosion Rate (mcd)			Solution Potential (Volts)
					3 hour	Exposure 7 day	14 day	
70391-1	64-64 Fe	24-46 C	135 gms.	800°C.	2.86			1.657
-2	64 Ave.	35 Ave.	135 "	"	?			1.658
-3			135 "	"		4.17		1.654
-4			135 "	"		<u>10.98</u>		1.656 Ave.
						7.07 Ave.		
70392-1	59-62 Fe	29-59 C	135 "	800°C.	29.0			1.649
-2	60 Ave.	43 Ave.	135 "	"	<u>10.1</u>			1.653
-3			135 "	"	19.5 Ave.	1.035		1.652
-4			135 "	"		<u>.411</u>		1.651 Ave.
						.723 Ave.		
70393-1	57-68 Fe	14-114 C	135 "	800°C.	4.87			1.655
-2	62 Ave.	61 Ave.	135 "	"	2.43			1.655
-3			135 "	"	<u>3.65</u> Ave.	.759		1.658
-4			135 "	"		<u>.526</u>		1.656 Ave.
						.642 Ave.		
70394-1	64-97 Fe	7-80 C	135 "	800°C.	7.59			1.655
-2	80 Ave.	43 Ave.	135 "	"	<u>.880</u>			1.660
-3			135 "	"	Ave.	1.62		1.659
-4			135 "	"		<u>.990</u>		1.658 Ave.
						1.30 Ave.		
<u>Pure Mg-C Series</u>								
70398-1	7-7 Fe	59-148 C	135 "	800°C.	1.70			1.668
-2	7 Ave.	103 Ave.	135 "	"	145.0			1.670
-3			135 "	"		4.59		1.668
-4			135 "	"		10.2		1.669 Ave.
70399-1	7-10 Fe	21-25 C	135 "	800°C.	.872			1.670
-2	8 Ave.	23 Ave.	135 "	"	<u>1.05</u>			1.667
-3			135 "	"	.96 Ave.	.410		1.669
-4			135 "	"		<u>.358</u>		1.669 Ave.
						.384 Ave.		
70400-1	12-18 Fe	16-39 C	135 "	800°C.	1.30			1.669
-2	15 Ave.	27 Ave.	135 "	"	<u>1.05</u>			1.669
-3			135 "	"	1.17 Ave.	.413		1.669
-4			135 "	"		<u>.373</u>		1.669 Ave.
						.393 Ave.		
70401-1	8-14 Fe	13-37 C	135 "	800°C.	.685			1.671
-2	11 Ave.	25 Ave.	135 "	"	<u>.619</u>			1.668
-3			135 "	"	.652 Ave.	.431		1.669
-4			135 "	"		<u>.378</u>		1.669 Ave.
						.404 Ave.		

TABLE II (3)

Alloy No.	Composition (ppm)		Melt Size	Thermal History	Alternate Immersion Corrosion Rate (mcd)			Solution Potential (Volts)
					3 hour	Exposure 7 day	14 day	
70402-1	5-6 Fe	<10-50 C	135 gms.	800°C.	.497			1.670
-2	5 Ave.	25 Ave.	135 "	"	1.24			1.670
-3			135 "	"	.87 Ave.	.380		1.669
-4			135 "	"		.337		1.670 Ave.
						.358 Ave.		
<u>Mg-Fe-Si Series</u>						<u>1 day</u>		
70433-1	22-23 Fe	<10 Si	3 Si*	135 gms.	800°C.	1.62		1.662
-2			135 "	"	1.50			1.661
-3			135 "	"	1.59 Ave.	.505		1.661
-4			135 "	"		.476		1.661 Ave.
						.490 Ave.		
70434-1	22-25 Fe	1900 Si	1490 Si*	135 "	800°C.	2.69		1.661
-2			135 "	"	3.26			1.661
-3			135 "	"	2.97 Ave.	31.4		1.661
-4			135 "	"		48.7		1.661 Ave.
						40.0 Ave.		
70435-1	19-20 Fe	370 Si	490 Si*	135 "	800°C.	1.37		1.662
-2			135 "	"	1.75			1.662
-3			135 "	"	1.56 Ave.	1.94		1.663
-4			135 "	"		2.34		1.662 Ave.
						2.14 Ave.		
70436-1	19-22 Fe	210 Si	250 Si*	135 "	800°C.	2.30		1.663
-2			135 "	"	2.12			1.663
-3			135 "	"	2.21 Ave.	2.53		1.662
-4			135 "	"		1.24		1.663 Ave.
						1.88 Ave.		
70437-1	22-26 Fe	140 Si	170 Si*	135 "	800°C.	1.43		1.663
-2			135 "	"	1.87			1.663
-3			135 "	"	1.65 Ave.	.467		1.663
-4			135 "	"		.530		1.663 Ave.
						.498 Ave.		
70438-1	70-76 Fe	60 Si	60 Si*	135 "	800°C.	170		1.646
-2			135 "	"	155			1.646
-3			135 "	"	162 Ave.	destroyed		1.645
-4			135 "	"		destroyed		1.646 Ave.

* Chemical analysis

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TABLE II (4)

Alloy No.	Composition (ppm)			Melt Size	Thermal History	Alternate Immersion Corrosion Rate (mcd)		Solution Potential (Volts)
						3 hour	Exposure 1 day	
70439-1	77-83 Fe	2300 Si	1480 Si*	135 gms.	800°C.	21.2		1.661
-2				135 "	"	27.6		1.659
-3				135 "	"	24.4 Ave.	157.7	1.660
-4				135 "	"		190.0	1.660 Ave.
							172.0 Ave.	
70440-1	74-79 Fe	560 Si	540 Si*	135 "	800°C.	107		1.654
-2				135 "	"	62.5		1.648
-3				135 "	"	85 Ave.	66.5	1.649
-4				135 "	"		61.0	1.647 Ave.
							114 Ave.	
70441-1	77-80 Fe	130 Si	130 Si*	135 "	800°C.	125		1.649
-2				135 "	"	124		1.651
-3				135 "	"	125 Ave.	destroyed	1.652
-4				135 "	"		destroyed	1.651 Ave.
70442-1	74-76 Fe	90 Si	90 Si*	135 "	800°C.	141.5		1.648
-2				135 "	"	133.5		1.649
-3				135 "	"	137 Ave.	destroyed	1.649
-4				135 "	"		destroyed	1.649 Ave.

TABLE III
SUBLIMATION DATA AND OPERATING CONDITIONS

Sublimation Run No.	Alloy No. of Sublimate	Alloy No. of Billet	Sublimations Received	Charge Weight (lbs.)	Charge Metal	Subl. Wt.Lbs.	Billet Wt.Lbs.	Scrap Subl. (lbs.)	Unsubl. Charge (lbs.)	Pressure		Temp. °C.		Hrs. at Temp.	Residue Wt.gms.	Residue \$ by Wt.	Residue Non Alloy No.	Filter	Remarks
										Microns Pirani	mm Hg. McLeod	Sub- limer	Con- denser						
SVC-13	--	67536	1	69	PM-DC(69782)	--	49	15	5	33	.001- .002	700	300- 350	144	28	.09	6396	Graphite Sieve with Graphite Granules	Cast at 825°C. for 5 hrs.
SVC-14	--	67537	2	45	67536	--	25.5	2	17.5	27-50	.002	700	300- 340	71	--	--	--	Graphite Sieve with Graphite Granules	Cast at 825°C. for 15 hrs.
SVC-15	--	67538	3	23	67537	--	8	9	6	22-23	.0007	675	300- 325	76	1.7	.02	--	Graphite Sieve with Graphite Granules	Cast at 825°C. for 15 hrs.

EFFECT OF IRON ON THE CORROSION RATE OF SUBMERGED MAGNESIUM
3 HOUR ALTERNATE IMMERSION EXPOSURE TO 3% NaCl

EFFECT OF IRON ON THE CORROSION RATE OF SUBMERGED MAGNESIUM
 3 HR ALTERNATE IMMERSION EXPOSURE TO 3% NaCl
 "AS CAST" "ANODIZED" SPECIMENS
 BASE ALLOYS - # 6751/5, # 6859
 WATER QUANTITY - COOLING RATE 11000°F/minute

FIG. 2

ALLOY NO.
 10655-70667
 70673, 70674

CE M
 3-22-52

MAGNESIUM-IRON CORROSION SPECIMENS
3 HOUR ALTERNATE IMMERSION IN 3% NaCl

COOLING RATE 11000° F/MINUTE

Alloy #70661-70674

Iron Content PPM

7

2.8 MCD

4.43 MCD

2.4 MCD

9

2.74 MCD

2.18 MCD

3.4 MCD

31

3.5 MCD

2.7 MCD

2.7 MCD

52

2.3 MCD

2.59 MCD

2.74 MCD

70

2.94 MCD

4.43 MCD

2.78 MCD

91

3.56 MCD

4.58 MCD

4.01 MCD

99

4.93 MCD

4.97 MCD

4.91 MCD

FIG. 4

MAGNESIUM-IRON CORROSION SPECIMENS
3 HOUR ALTERNATE IMMERSION IN 3% NaCl
COOLING RATE 1000° F/MINUTE

Iron Content PPM

Alloy #70661-70674

127

7.25 MCD

8.95 MCD

8.15 MCD

135

6.4 MCD

11.3 MCD

4.93 MCD

184

15.47 MCD

17.7 MCD

23.6 MCD

205

4.97 MCD

3.29 MCD

5.3 MCD

235

14.49 MCD

7.11 MCD

22.13 MCD

275

15.02 MCD

22.45 MCD

5.35 MCD

310

9.0 MCD

17.3 MCD

FIG. 5

MAGNESIUM-IRON CORROSION SPECIMENS
 1 DAY ALTERNATE IMMERSION IN 3%NaCl
 COOLING RATE 11000° F/MINUTE
 Alloy #70655-70674

Iron Content (PPM) Corrosion Rate (MCD) Iron Content (PPM) Corrosion Rate (MCD)

7		.708	127		7.91
9		.521	135		10.4
31		.635	184		19.7
52		1.09	205		24
70		1.14	235		58.7
91		1.47	275		104.1
99		2.16	310		19.99

FIG. 6

MAGNESIUM-IRON CORROSION SPECIMENS

ALTERNATE IMMERSION IN 3% NaCl

99 PPM IRON

Alloy #70662-70671

3 HOUR EXPOSURE

SLOW COOLED (50° F/MINUTE

180.3 MCD

129.9 MCD

147.0 MCD

RAPIDLY COOLED 11000° F/MINUTE

4.93 MCD

4.97 MCD

4.91 MCD

1 DAY EXPOSURE

266 MCD

2.16 MCD

FIG. 7

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MAGNESIUM IRON CORROSION SPECIMENS
1 DAY ALTERNATE IMMERSION IN 3% NaCl
(~175 ppm Fe)

ENTIRELY SOLIDIFIED
WATER QUENCHED

ALLOY #70863

ALLOY #70864

PARTIALLY SOLIDIFIED
WATER QUENCHED

ALLOY #70865

ALLOY #70866

FIG. 8

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EFFECT OF IRON ON THE SOLUTION POTENTIAL OF PURE MAGNESIUM

NOTE:
ALLOYS COOLED AT 4000°F
FROM PARTIALLY SOLIDIFIED STATE

ALLOY
70655-70674

QCM
2.1-3.5

FIG 9.

43

KEUFFEL & ESSER CO., N. Y. NO. 389-120
Logarithmic, 8 X 8 Cycles.
MADE IN U. S. A.

44

EFFECT OF CARBON ON THE CORROSION RATE OF PURE MAGNESIUM
CONTAINING ~20 PPM IRON - ALTERNATE IMMERSION IN 3% NaCl

45

EFFECT OF CARBON ON THE CORROSION RATE OF PURE MAGNESIUM
CONTAINING ~ 66 PPM IRON ALTERNATE IMMERSION IN 3% NaCl

46

○ - MG + 9 PPM Fe
● - MG + 20 PPM Fe
□ - MG + 66 PPM Fe

ALLOY
Z 70385-70402

FIG. 13

ccm.

3-27-52

47

EFFECT OF SILICON ON THE CORROSION RATE OF SUBLIMED Mg. (6 PPM Fe)

FIG. 14

0.3% Fe EXPOSURE
 1 DAY EXPOSURE

ALLOY NO.

68270-68276

CEM

3-19-52

KEUFFEL & ESSER CO., N. Y. NO. 358-120
Logarithmic 3 X 8 Cycles
MADE IN U.S.A.

19-26 PPM Fe
22 PPM AVE.

KEUFFEL & ESSER CO., N. Y. NO. 389-120
Logarithmic, 3 X 3 Cycles.
MADE IN U.S.A.

70-83 PPM Fe
77 PPM AVE.

50

51

52

53

54

EFFECT OF CHLORIDE CONTENT ON THE CORROSION RATE OF HIGH PURITY MAGNESIUM
7 DAY EXPOSURE TO 3% NaCl

ALLOY #
68409 - 68415
R.W.F. JR
3/12/51

FIG. 21

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ARC-MELTING POT

FIGURE 23

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Cooling Rate, Sublimed and Argon - Cast Magnesium.

Figure 24

Figure 25

Photomicrograph of Triply-Sublimed
and Arc-Melted Magnesium

750X

Alloy No. 69629-4
Electrolytically Polished