WADC TECHNICAL REPORT 52-41





# TERNARY MAGNESIUM-LITHIUM BASE CONSTITUTION DIAGRAMS AND MAGNESIUM ALLOYS OF LOW ALLOY ADDITIONS

One of a series of reports
on
MAGNESIUM ALLOY RESEARCH

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*MARCH 1951*

WRIGHT AIR DEVELOPMENT CENTER

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MAGNESIUM ALLOY RESEARCH

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*Rensselaer Polytechnic Institute*

*March 1951*

*Materials Laboratory*
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Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

## FOREWORD

This report was prepared by the Rensselaer Polytechnic Institute, Troy, New York, under Supplemental Agreement No. 1 (S-50-1021) of Contract No. W33-038-ac 22542 (21337), under the Research and Development Order No. R605-230. This work was administered under the direction of the Materials Laboratory, Research Division, Wright Air Development Center, with George W. Orton, Capt., USAF, and Frederick C. Krug, Capt., USAF, acting as project engineers.

## ABSTRACT

This report summarizes the experimental work on the study of two independent subjects.

The first part of the report describes the development and application of techniques for establishing phase boundaries in the magnesium-lithium-aluminum and magnesium-lithium-zinc ternary systems. Based primarily on microscopic examination, the tentative locations of phase boundaries at 700°F are presented for these two systems. Additional work at 700°F and also at 500°F is in progress.

The second part of the report summarizes the progress in experimental development of magnesium-base alloys with low alloy additions. The primary purpose of this investigation is to obtain alloys having a favorable combination of medium strength and high formability. A major portion of the development work was devoted to a continued study of magnesium-zinc-cerium alloys. Information is presented to demonstrate that an attractive combination of mechanical properties may be obtained over a reasonably wide range of zinc and cerium concentrations. Warm rolling of sheet, followed by a stress relieving heat treatment, produced the most favorable mechanical properties. Details of two successful procedures for introducing zirconium in magnesium are described.


## PUBLICATION REVIEW

Manuscript Copy of subject report has been reviewed and is approved for publication.

FOR THE COMMANDING GENERAL:

M. E. SORTE
Lt. Colonel, USAF
Chief, Materials Laboratory
Research Division

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# INTRODUCTION

The activities of this research program were directed toward a study of two individual problems:

1. Location of phase boundaries at various temperature levels in the solid state for the magnesium corner of the magnesium-lithium-aluminum and magnesium-lithium-zinc systems.
2. Development of magnesium-base alloys with a relatively low total alloy addition for the purpose of providing a combination of good formability, toughness and moderate strength.

The locations of alpha and beta phase boundaries and the identification of phases present in adjacent phase fields as influenced by temperature changes in the solid state were topics of principal interest in the phase boundary study. Initial work described in this report was at the 700°F and 500°F temperature levels. The experimental methods selected for establishing phase boundaries were microscopic examination, electrical resistivity measurements at the elevated temperature of interest and x-ray diffraction at the elevated temperature of interest. The most formidable problem encountered in the investigation was the prevention of a loss of lithium from the surface of specimens and a general reaction between the specimen and its surrounding atmosphere during long holding periods at 700°F required to approach equilibrium conditions. Some progress was made in minimizing the effects of these conditions at 700°F. This was

accomplished at the expense of a major share of the time spent on the problem. Microscopic examination was the most immediately applicable technique and was the method used in obtaining the results presented in this report.

A limited amount of previous work had indicated the possibility of an improvement in ductility and toughness of magnesium base alloys with small amounts of alloy additions (called dilute alloys for brevity) compared with present day commercial alloys. The basis for proceeding on the problem was the belief that a favorable combination of ductility and strength would be obtained with some dilute composition containing one or more of the elements zinc, cerium, zirconium, calcium, titanium and possibly nickel, copper and thallium. In addition, it was believed that the maximum capabilities of these dilute alloys would be achieved with some combination of hot, warm, straight and cross-rolling procedure, followed by a heat treatment to produce an extremely fine, equi-axed recrystallized grain structure. Encouraging results with an alloy nominally magnesium-0.8 zinc-0.2 cerium led to the more detailed investigation described in this report of variations in composition and thermal treatment for alloys in this system.

## CONCLUSIONS

1. Phase Boundary Study

   a. The phase boundary locations determined by the microscopic examination method at the 700°F isothermal for the magnesium-lithium-aluminum and magnesium-lithium-zinc systems were considered tentative, subject to verification by more detailed study. The tentative evidence, however, indicated an extensive range of the alpha and particularly the beta phase field in the magnesium-lithium-zinc system. These phase fields were more restricted in the magnesium-lithium-aluminum system.

   b. The most effective method used to minimize the conditions of a loss of lithium and a general surface reaction of the specimen with the surrounding atmosphere during extended periods at 700°F consisted of a thorough mechanical abrasion of the extruded surface of the sample and protection with double charcoal refined, welding grade helium.

   c. The extent of the effects of loss of lithium and surface attack of samples heated at 500°F or below was not significant. No investigation was made of temperatures between 500 and 700°F to establish the threshold temperature at which these effects became appreciable.

2. Dilute Alloy Study

a. A combination of relatively high strength properties and elongations exceeding 20% in 2 inches was obtained for a group of magnesium-zinc-cerium alloys in a region of concentrations ranging from approximately 0.7 to 1.3% zinc and 0.25 to at least 0.4% cerium.

b. The most beneficial sheet preparation was consistently the warm rolled and stress relieved procedure. This combination produced an extremely fine, equi-axed grain structure.

c. Two procedures were used successfully to prepare magnesium-zirconium binary alloys. These methods introduced zirconium as dense zirconium tetrachloride and as zirconium sponge. The maximum concentration obtained was 0.76% zirconium.

## RECOMMENDATIONS

1. Phase Boundary Study

   a. It is recommended that the location of phase boundaries by the microscopic method be continued at 700 and 500, and extended to the 300, 200 and 150°F temperature levels in the magnesium-lithium-aluminum and magnesium-lithium-zinc systems.

   b. Continuation of development of the techniques of electrical resistivity and x-ray diffraction is recommended to provide supplementary techniques for phase boundary locations.

2. Dilute Alloy Study

   a. Additional work on alloys in the magnesium-zinc-cerium system is recommended in view of the promising mechanical properties exhibited by some alloys.

   b. A survey of the mechanical properties in the magnesium-zirconium and magnesium-zinc-zirconium systems is recommended.

# MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

## PHASE BOUNDARY STUDY

### Melting and Casting Magnesium-Lithium Base Ternary Alloys

Melting and Casting Technique

The design, construction and operation of equipment for melting and casting magnesium-lithium base alloys in a common atmosphere of argon was described in the Summary Report for 1949(1). This early melting and casting of magnesium-lithium base alloys for phase boundary studies was made in an apparatus constructed of plain carbon steel. It was found that non-metallic inclusions were present in ingot and extruded microstructures, being more pronounced in the higher lithium content alloys. Although the identity of the inclusions was not established, it was considered that the most probable source of this condition was direct pick-up from the surface of the plain carbon steel crucible, stir rod and mold of the apparatus. In order to minimize this condition, the equipment was reconstructed, using ferritic stainless steel for all parts exposed to liquid melt.

Two modifications of the original operating procedure were made. These were:

1. Superheat all melts to a minimum temperature of 1400°F
2. Use an increased length of settling period after the final stirring operation.

A detailed description of the experimental procedure for melting and casting magnesium-lithium base ternary alloys is given in Appendix I.

As a general observation, the alloys melted and cast with the ferritic stainless steel apparatus gave evidence of a consistent improvement in the degree of freedom from non-metallic inclusions compared with melts prepared in the plain carbon steel construction. Observations also indicated that the increased fluidity of the melt caused by a higher temperature of melting and the increased length of settling time resulted in an improvement in ingot quality.

Investigation of the Use of Flux in Melting Procedure

Two heats were made as the beginning in a study of the effectiveness of using a flux to promote better separation of metal and non-metallics. In this work ferritic stainless steel crucibles, molds and stirring rods were used in the equipment for melting and casting in an inert atmosphere. In both heats 50 grams of a lithium chloride-lithium fluoride mixture was used having a solidification range above the pouring temperature of the magnesium-lithium alloy. Intended compositions of the two alloys, the flux additions and the recovery of metal are given in Table I.

TABLE I

INTENDED COMPOSITIONS OF ALLOYS, FLUX MIXTURES AND METAL RECOVERY IN PRELIMINARY STUDY OF USE OF FLUX IN MELTING OPERATION

| Alloy | Intended Composition | | | Intended Flux Addition | | Metal Recovery |
|---|---|---|---|---|---|---|
| | Mg/Li | Mg | Li | LiCl | LiF | Percent |
| L-91 | 8.12 | 89.0 | 11.0 | 6 gm. | 44 gm. | 90.5 |
| L-92 | 9.0 | 90.0 | 10.0 | 10 | 40 | 92.8 |

The melting schedule for L-91 and L-92 was as follows:

1. Charge components in crucible, seal apparatus and flush with tank argon for 10 minutes.
2. Melt down under approximately 2 psi. pressure of argon.
3. When molten stir for 1 minute.
4. Superheat to 1450°F and stir for 1 minute.
5. Cool slowly in furnace to 1220°F and pour.

In each case the flux solidified above the pouring temperature of the alloy and remained in the crucible when the charge was poured. For alloy L-91, however, the flux appeared to be viscous and adhered to the stirring rod. It is believed that the higher range of LiCl (20%) was more useful than the lower range (12%).

On the basis of this experience six additional heats were prepared using 10 gm. of LiCl and 40 gm. of LiF as a flux. The procedure used in preparing these alloys was the same as was used in preparing L-91 and L-92. Table II contains a list of these alloys together with their intended compositions and the recovery of metal.

Microscopic examination of these alloys indicated that the addition of a flux reduced the number of non-metallic inclusions. Alloy L-161 was exceptionally clean in comparison with other alloys of similar compositions. The remainder of the alloys compared favorably with the best alloys prepared without a flux. Alloy L-160 was superheated to a temperature of 1520°F, but the increase in temperature did not produce a cleaner structure. As

TABLE II

INTENDED COMPOSITION OF ALLOYS AND METAL RECOVERY PREPARED WITH A 10 LITHIUM CHLORIDE-40 LITHIUM FLUORIDE FLUX

| Alloy | Intended Composition Mg/Li | Mg | Li | Zn | Metal Recovery Percent |
|---|---|---|---|---|---|
| L-116 | 30 | 87.1 | 2.9 | 10.0 | 86.1 |
| 117 | 30 | 82.3 | 2.7 | 15.0 | 86.1 |
| 118 | 30 | 77.4 | 2.6 | 20.0 | 92.0 |
| 160 | 8 | 71.1 | 8.9 | 20.0 | 88.0 |
| 161 | 10 | 81.8 | 8.2 | 10.0 | 93.6 |
| 162 | 10 | 80.0 | 8.0 | 12.0 | 87.2 |

a disadvantage, the cleaning of the crucibles after pouring was difficult when flux additions were made. It is believed, however, that this disadvantage could be overcome to some extent by re-heating the crucibles and pouring out most of the flux. This procedure was not investigated. It was decided that the additional time involved in preparing a flux heat and in cleaning the apparatus after completing the heat was not justified by the general order of improvement resulting from the treatment. This decision was influenced considerably by the consistent improvement in ingot quality resulting from using ferritic stainless steel equipment, using practices favoring better separation of non-metallics from the melt and from more experience in the technique of preparing the alloys.

## MAGNESIUM-LITHIUM BASE TERNARY ALLOYS PREPARED

### Selection of Compositions

An initial series of magnesium-lithium-aluminum and magnesium-lithium-zinc alloys was selected arbitrarily near anticipated locations of phase boundaries. As evaluation of compositions proceeded, new compositions were selected closer to phase boundaries.

### Alloy Charging Components

Alloy charging components were the purest metals available. These metals are identified with respect to physical state and purity:

1. Magnesium - re-distilled, high purity crystals supplied by the Dow Chemical Company. The analysis of one shipment of this metal was as follows, weight percent:

| | | | | | |
|---|---|---|---|---|---|
| Al | 0.003 | Ni | 0.001 | Zn | 0.01 |
| Ca | 0.01 | Pb | 0.01 | Mn | 0.002 |
| Cu | 0.001 | Si | 0.01 | | |
| Fe | 0.001 | Sn | 0.001 | | |

2. Lithium - purified lithium ingot, supplied by Dow Chemical Company, Magnesium Division, Midland, Michigan. No analysis was furnished with the ingot shipment. The concentration of sodium, believed to be the principal impurity, was 0.02%, as determined by flame photometer analysis.

3. Aluminum - high purity notched pig, supplied by Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pennsylvania. Specified as 99.99% aluminum, the following analysis was supplied:

| | |
|---|---|
| Cu = 0.0038% | Mg = 0.0009% |
| Fe = 0.0007 | Na = 0.0001 |
| Si = 0.0014 | Ca = Not detected |
| Mn = Not detected | |

4. Zinc - Horsehead Special (99.99 + % Zn) pig, supplied by Belmont Smelting and Refining Works, Inc., 330 Belmont Ave., Brooklyn, N.Y. Guaranteed purity was:

Pb = 0.006% maximum

Fe = 0.005% maximum

Cd = 0.004% maximum

Sum of Pb, Fe and Cd 0.01%

Alloys Melted and Cast

A complete list of the intended compositions and chemical analyses, where determined, of all magnesium-lithium base ternary alloys melted and cast is given in Table XIX, Appendix II.

# EXTRUSION OF MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

## Purpose of Extrusion

Extrusion was selected as a means of mechanical deformation of experimental magnesium-lithium base alloys for the following reasons:

1. To provide a reproducible method of thoroughly working the cast structures under controlled conditions of deformation. It was anticipated that the uniformity of phase distribution and the eventual approach to equilibrium conditions would be enhanced by a reduction approaching 100%.
2. To provide a method of mechanical working of complex cast structures too brittle to work by rolling, forging, swaging or drawing.

## Homogenization of Billets for Extrusion

Prior to extrusion of 1-1/2 inch diameter ingots to 1/8 in. diameter rod, it was a standard practice to homogenize the ingots 48 hours at 500°F. The ingots were given several coats of Dow-Corning 993 silicone varnish (50% solids) before heating. This method of surface protection was not entirely successful and in some cases the ingots were severely oxidized at sites of breakdown of the coating. It was necessary to discard these alloys and to develop a more suitable means of protection during the cycle.

The possibility of using an English proprietary mixture

"Keepbryte"(a) as a means of providing protection was investigated. This material produced an extremely adherent, glass-like surface coating at 500°F. The mixture was applied to ingots that were preheated for 10 minutes in an electric resistance furnace controlled at 500°F. This length of time was sufficient to heat the ingots to a temperature that would fuse the "Keepbryte" and form an adherent coating. The ingots were then returned to the furnace for a 48 hour treatment at 500°F. Experience with this material indicated that excellent surface protection was given to most magnesium-lithium base alloys homogenized 48 hours at 500°F. It was found, however, that alloys containing large amounts of both lithium and zinc (greater than 10%) were not protected from attack by the mixture. These same alloys suffered severe damage at 500°F when coated with Dow Corning 993 silicone varnish prior to homogenization.

As a result of these observations, it was decided to use "Keepbryte" for surface protection during homogenization of all magnesium-lithium base alloy billets except those containing both lithium and zinc in concentrations greater than 5% of each component. It is planned to homogenize future high lithium-high zinc alloys after sealing each alloy individually in a tightly capped steel pipe.

Experimental Procedure for Extrusion

The extrusion of magnesium-lithium base alloys was carried out by the direct method, using a small laboratory-scale

(a) Trade name of a material supplied by Kasenit Lt'd., 7 Holyrood Street, London S.E. 1. The material was reported to consist of essentially boric acid with a small percentage of ferric oxide.

extrusion press. Originally, the equipment was mounted in a Southwark-Emery testing machine having a maximum available force of 50 tons. The equipment was modified to permit operation on a 125 ton Watson-Stillman hydraulic press, resulting in the following improvements:

1. The greater available force permits extrusion of compositions which could not be extruded in the 50 ton capacity arrangement.
2. Lower extrusion temperatures are permitted.
3. There has been a significant improvement in surface quality and straightness of the 1/8" diameter extruded rod.

A standardized procedure was established whereby the alloys were extruded to 1/8 in. diameter rod at the lowest temperature possible with the force available using an extrusion ratio of 144 to 1 - a reduction in cross-sectional area of over 99%. The process of extruding experimental alloys was accelerated by preheating billets in a muffle furnace controlled at 500°F for 10 minutes prior to charging for extrusion.

A summary of the extrusion conditions for all magnesium-lithium base ternary alloys extruded for phase boundary study is given in Table XIX, Appendix II.

## CHEMICAL ANALYSIS OF MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

### Methods of Quantitative Analysis

Procedures used for the quantitative determination of lithium, aluminum and zinc in magnesium-lithium base alloys were obtained from the Dow Chemical Company. Initial determinations of lithium were made with a gravimetric method. Later, a Perkin-Elmer Flame Photometer, Model 52A, was obtained and a majority of the lithium determinations reported were made by this instrument. Occasional checks by the gravimetric method were made, showing close agreement between the two methods. Determinations of aluminum and zinc were by gravimetric methods.

A summary of all quantitative determinations of lithium, aluminum and zinc is given in Table XIX, Appendix II.

## METALLOGRAPHIC STUDY OF MAGNESIUM-LITHIUM BASE ALLOY PHASE BOUNDARIES

### General Plan and Procedure for Study

Experimental work on the application of the microscopic method of locating phase boundaries in the magnesium-lithium-aluminum and magnesium-lithium-zinc systems was emphasized almost to the exclusion of the x-ray diffraction and electrical resistivity methods. It was considered probable that the microscopic method would be the most readily adaptable and informative of the three methods for initial locations of phase boundaries. The principal application for the other two methods was to supplement and possibly confirm microscopic data, providing auxiliary methods to investigate controversial details.

The general procedure of the microscopic examination method used in this initial phase boundary work was:

1. Prepare samples of 1/8 in. diameter rod extruded from ingots homogenized at 500°F to a reduction in cross-sectional area of over 99% at the lowest temperature permitted by the available force of the extrusion equipment.
2. Heat samples at 700°F and 500°F temperature levels to achieve equilibrium conditions, using a protective atmosphere to minimize attack of samples at these elevated temperatures. A sufficient length of sample was used to provide material for microscopic examination and chemical analysis.

3. Quench rapidly to room temperature in kerosene.

4. Mount transverse and longitudinal sections of the heat treated rod in a plastic material at room temperature and without pressure to minimize the possibility of destruction of the elevated temperature conditions in the quenched samples.

5. Examine the microstructures and with the aid of suitable etchants determine phases present and relative amounts.

6. Determine by chemical analysis the concentration of lithium, aluminum or zinc in the samples found to be near phase boundaries.

7. On the basis of the microscopic examination and chemical analysis select new compositions of alloys closer to phase boundaries for preparation and evaluation.

Metallographic Technique

A detailed description of the metallographic procedures for magnesium-lithium base alloys is summarized in Appendix IV.

Development of Methods for Thermal Treatment of Alloys

The development of a successful procedure for obtaining, at room temperature, a structure representative of high temperature equilibrium in magnesium-lithium base alloys was a major problem in the application of the microscopic technique to the phase boundary study. Three individual approaches to the problem of sample protection during heat treatment were:

1. Design and construction of a specimen container to hold a number of specimens in an inert atmosphere in order to

attain equilibrium conditions at elevated temperatures. An important requirement of the design was to permit rapid transfer of samples from the fixture to a quenching medium.

2. Development of a procedure to seal samples in Pyrex tubes for protection during heat treatment.

3. Evaluation of the use of a proprietary mixture "Keepbryte" (see the discussion on homogenization of ingot structures p. 7 ) for surface protection during heat treatment.

A discussion of the development of each method is given below.

1. Development of a Specimen Container for Heat Treatment

Initial work on this development was described in the last Summary Report[1]. Experimental work on the original holder machined from a block of aluminum alloy 2S was discontinued after repeated efforts to obtain an effective seal for the protective atmosphere were unsuccessful.

The design of a specimen holder was changed to provide chambers for 16 samples in a single, cored and cast block of Alcoa #356 aluminum alloy. An inert atmosphere was distributed to each chamber by means of a common manifold, cored into the cast block. A single seal to retain the specimens and the protective atmosphere was provided. An assembly drawing of the specimen

container is shown in Fig. 1. The bottom of the container was sealed with a single gasket held between the container and the bottom by a steel retainer plate. Rapid ejection of the specimens was accomplished by removing a steel wedge to release the latch holding the retainer plate. Specimen ends were seated in small graphite spacers to prevent physical contact with the chamber walls and to facilitate rapid removal during the quenching operation.

An electrical resistance furnace was constructed for use with the container. The furnace core, an alundum tube 5" inside diameter and 12" long, was mounted vertically in an insulated steel shell with provision for openings at both ends. The specimen container assembly was suspended in the furnace tube by means of a steel frame. At the completion of the heat treatment cycle the furnace bottom was removed and the specimen container was lowered to a position where it was opened to discharge specimens into a quenching bath.

Initial operation of the container in 96 hour runs at 700°F, using tank helium as a protective atmosphere, indicated that a general surface attack of specimens was related to the rate of flow of the helium into the container. A more severe attack with a greater flow of helium was considered evidence of the action of a minor extent of impurities in the gas. On the basis of these

## Key to Details in Figure 1

1. Specimen Container, a single casting, Alcoa 356 alloy.
2. Atmosphere Inlet, 1/8" standard steel pipe.
3. Specimen Chambers, 16 holes 9/32" diameter, 2" deep, 13/32" between centers.
4. Thermocouple Well, 5/32" diameter, 2" deep.
5. Atmosphere Distribution Manifold, 2" x 1-3/4" x 1/2", 3/8" radii at corners, 1/2" diameter center bulb.
6. Wedge Back-Up Plate, stainless steel, 2-7/8" x 3" x 1/8", secured with six No. 8-32 x 1/2" long stainless steel machine screws.
7. Gasket, copper-asbestos sandwich type.
8. Container Bottom Block, Alcoa 356 alloy, 2-1/2" x 2" x 1/2".
9. Latch Shaft, drill rod, 3/16" diameter x 3" long.
10. Latch Wedge, stainless steel, 3" x 3/4" x 1/8".
11. Bottom Retainer Plate, stainless steel, 2-7/8" x 2-3/8" x 1/8"; position adjusted with six No. 8-32 x 1/2" long machine screws.
12. Latch, stainless steel.

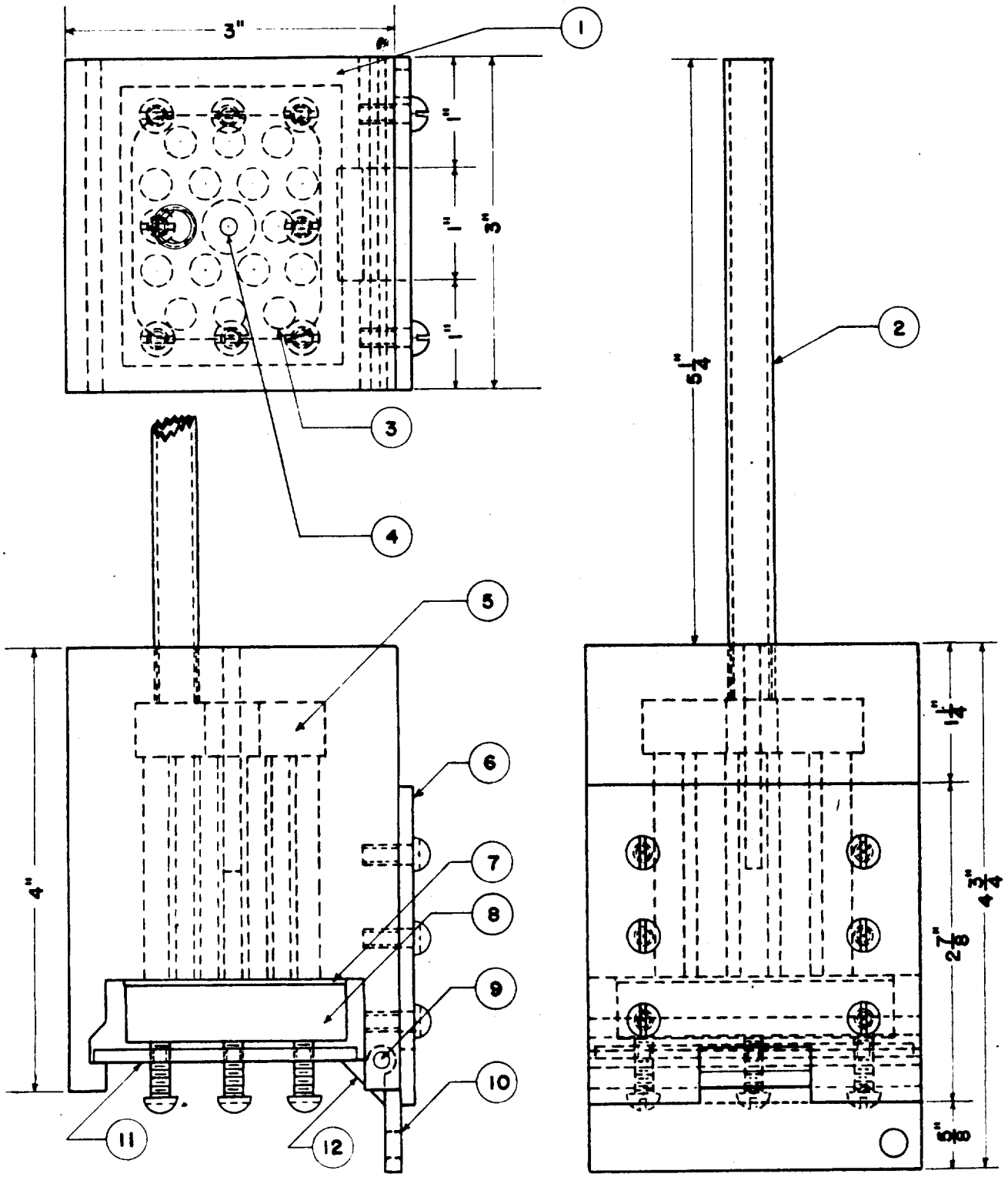


FIG. 1

SPECIMEN CONTAINER FOR HEAT TREATING MAGNESIUM-LITHIUM BASE TERNARY ALLOYS FOR MICROSCOPIC EXAMINATION SCALE = 3/4 SIZE

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observations a purification train was assembled and in operation the rate of flow of the inert gas was adjusted to a minimum.

The train consisted of (1) a mercury seal safety valve on the input end of the train leading from the tank pressure regulator, (2) a Milligan bottle containing concentrated sulfuric acid, (3) a tube furnace operating at 1200°F containing calcium chips, (4) a second tube furnace operating at 320°F containing lithium foil, and, (5) a mercury manometer on the output end of the train leading to the specimen container. Comparison of samples heat treated in the aluminum container with and without the gas purification train indicated that the train had little effect upon the amount of surface attack. The use of the train was discontinued.

Continued operation of the container for heat treatment of samples, using tank helium as an atmosphere indicated that results comparable to the method of sealing samples within Pyrex tubing were obtained.

Repeated use of the apparatus at 700°F caused excessive distortion of the aluminum casting in the region of the latch mechanism and eventually prevented rapid release of heat treated samples. It is believed that this problem would be minimized by the use of steel in all highly stressed members of the apparatus. Work on this equipment was halted temporarily because it was

found that the method of sealing samples in Pyrex for protection during heat treatment offered an immediate and acceptable solution to the problem of preparing samples for microscopic study.

2. Development of Procedure to Seal Samples in Pyrex Tubes for Protection During Heat Treatment

A procedure was developed to protect magnesium-lithium base alloys during heat treatment by sealing within Pyrex tubing. Samples of 1/8 in. diameter extruded rod approximately 3 in. long were cut, identified by stencilling and placed within 1/4 in. inside diameter thin wall Pyrex tubing. Physical contact between the samples and the tubing was prevented by means of a small washer on each end machined from spectrographic carbon electrodes. A thin, spherical bulb, approximately 1/2 in. diameter was blown on one end of the Pyrex tube to facilitate fracturing and rapid ejection of the specimen during quenching. A photograph of a Pyrex tube, sample and washers and a completed sample sealed within the tube is shown in Fig. 2.

A preliminary investigation was made to determine the type of atmosphere to seal within the tubes in order to obtain the most protection during heat treatment. Three types of atmospheres were studied:

a. The tube was evacuated by means of a small mechanical vacuum pump. The exact magnitude of the pres-

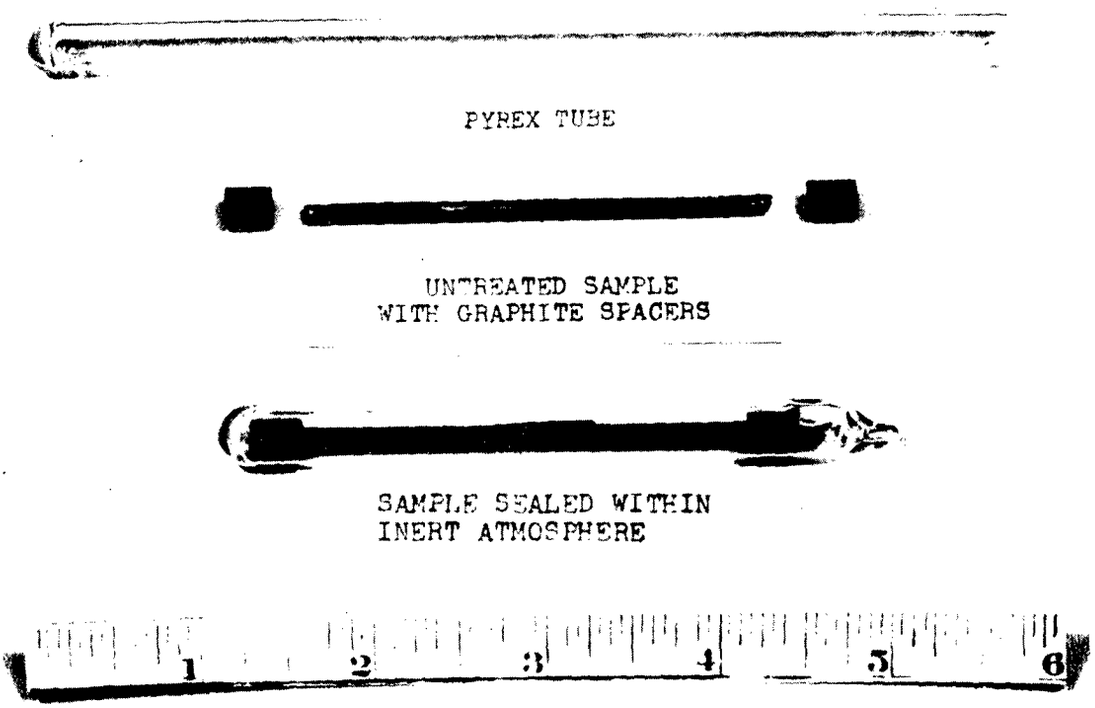



Figure 2

Illustration of the Pyrex tube blank, sample and end washers and completed sample sealed in tube for protection during heat treatment of magnesium-lithium alloys.

sure was not measured but was estimated to be of the order of 0.1 mm. of mercury.

b. The tube was alternately evacuated and flushed with tank argon for a series of three cycles and eventually sealed to leave a slight positive pressure of argon within the tube.

c. The same general procedure as for argon was applied using double charcoal refined helium of a guaranteed purity of 99.99% helium.

The results of this study using alloy L-21 (magnesium-11.85% lithium) and a heat treatment of 24 hours at 700°F indicated that the helium atmosphere provided better sample protection than the other two methods. The least protection was provided by the evacuated atmosphere.

The following standard procedure for sealing samples within Pyrex was then established and used in all routine heat treatment of magnesium-lithium-aluminum and magnesium-lithium-zinc alloys.

a. Use samples of 1/8 in. diameter extruded rod 3 in. long. This sample length provided ample material for a transverse and longitudinal section for microscopic examination and for chemical analysis after heat treatment.

b. Insert sample with graphite washers on ends into tube. Alternately evacuate and flush with helium

three times.

c. Seal the assembly under a slight positive pressure of helium within the tube.

3. Evaluation of Sample Protection Afforded by "Keepbryte"

As a result of the favorable action of the proprietary compound "Keepbryte" in protecting billets during homogenization at 500°F prior to extrusion, an auxiliary investigation was made to determine the suitability of this material for protecting 1/8" diameter extruded rod specimens at 700°F in preparation for microscopic examination. An alloy particularly susceptible to damage during heat treatment at 700°F was selected for trial (alloy L-90, charge 223, magnesium-11.4 lithium-20.0 zinc).

Six specimens were heated to approximately 400°F and plunged into "Keepbryte" powder. This operation was repeated four times in the hope that the coating would be more continuous. The specimens were then heated 24 hours at 700°F in air and quenched in kerosene. It was found that the coating had not provided complete protection and that attack of the specimens proceeded inward from these sites of coating breakdown.

No additional work with "Keepbryte" during heat treatment of specimens for microscopic examination was carried out.

Selection of Method for Sample Protection During Heat Treatment

The method of protecting magnesium-lithium alloys by sealing within Pyrex tubing was selected as the standard procedure and practically all alloys examined microscopically were processed by this method. This decision was based to a considerable extent on the fact that the Pyrex tubing method was satisfactory and could be applied immediately without additional development to accelerate the heat treatment program. The tedious nature of the sealing process compared to the aluminum container method was not a serious obstacle in practice.

A comparable degree of sample protection was provided by the Pyrex tubing procedure and the aluminum container. Both methods were successful for practically all ranges of compositions studied in the two ternary systems for temperatures up to 500°F. Heat treatment of specimens at 700°F resulted in a consistent observation of a depletion of the beta phase in the surface of specimens and a sub-surface reaction appearing much like an internal oxidation. The depth of penetration of these conditions was more pronounced the higher the concentration of lithium and the longer the time of heat treatment.

In most samples of alloys heat treated by either procedure at 700°F, the center structure was not damaged by the treatment. The microscopic examination and chemical analysis of this structure was the basis for proceeding on the metallographic portion of the investigation of phase boundaries. It was disturbing, however, to know that these surface conditions result to a signif-

icant degree in the alloys containing a large amount of lithium and zinc or lithium and aluminum. A considerable amount of time was spent in an effort to find the cause of the trouble and to overcome the difficulty.

More recent information on the optimum procedure for using helium as a protective atmosphere, obtained in connection with development of the electrical resistivity apparatus for phase boundary study (see p. 62), indicated that an improvement in the resistance to surface reaction was provided by polishing the sample with fine emery paper prior to heat treatment in a helium atmosphere. This may indicate that the problem of surface attack is related to conditions existing on the extruded surface of the sample prior to heat treatment. Work on this aspect of the problem is in progress.

Experimental Procedure for Heat Treatment of Samples for Microscopic Study

Samples of magnesium-lithium base alloys sealed in Pyrex tubing were heat treated for microscopic study in an electrical resistance furnace. The furnace temperature was controlled with a Wheelco "Capacitrol" Model 224.

During heat treatment, Pyrex tubes were held in a massive block of aluminum in order to minimize temperature fluctuations. The block accomodated 14 tubes. A chromel-alumel thermocouple was passed through a small hole in the door of the furnace and its hot junction was held rigidly against the aluminum block. The chromel-alumel thermocouple was calibrated against a platinum,

platinum-rhodium thermocouple standardized by the National Bureau of Standards. Sample temperatures were measured with a Brown Potentiometer, Model 1117. When the furnace was controlling at the 700°F temperature level the average sample temperature was 695°F, with a maximum observed temperature variation of plus or minum 7°F and an average variation of plus or minus 4°F. At the 500°F temperature level the average sample temperature was 495°F, with maximum and average temperature variations approximately the same as at the 700°F level.

At the conclusion of the heat treatment the pyrex tubes were removed from the block, transferred rapidly to a quenching bath, fractured and dropped into the bath. Kerosene was used as the quenching medium for practically all samples. In several cases where water was used for quenching, a distinct reaction between the sample and water was observed and the use of water was abandoned. The kerosene bath was retained in a massive steel crucible and had a volume of approximately 1 liter. Pyrex tubes were crushed against the top flange of the crucible and specimens dropped into the kerosene. The average elapsed time between removing the tubes from the furnace and immersion in the kerosene was three seconds. The kerosene bath was used at room temperature.

From microstructural observations kerosene was found to be effective in preserving the phase relationships at the temperature of quenching. In a few isolated specimens, however, evidence of precipitation from solid solution was observed. An example of this condition is shown in Fig. 11 for alloy L-90 (magnesium-9.9%

lithium-20.2% zinc) heat treated 24 hours at 700°F and quenched in kerosene. In these cases it was suspected that the rate of quenching may have been retarded by the kerosene temperature rising to an appreciable degree above room temperature and to a delay in transfer from the furnace to the quenching bath. Subsequent study indicated that this suspicion was correct.

Tentative Determination of Sample Heat Treatment Time

Auxiliary metallographic studies were made to determine a tentative time for heat treatment of samples at 500°F and 700°F. The object of this work was to select a length of time to be used as a standard treatment of all samples, at a given temperature, such that conditions would be obtained to provide a close approach to equilibrium for the tentative location of phase boundaries in both systems studied.

A study was made to compare the degree of approach to equilibrium for samples heat treated 24 and 72 hours at 700°F, respectively. Alloys used for this work, together with treatments used and microstructural observations are listed in Table III. The longer time of treatment was carried out in Pyrex tubes and the shorter time of treatment in the aluminum container. Comparing the 72 hour and 24 hour heat treatment times, microstructures in the centers of specimens of alloys appeared practically identical with respect to relative percentages of phases present. A slightly greater degree of beta phase agglomeration and a small amount of grain coarsening was evident after 72 hours. An example of these observations is given in Figs. 3, 4 and 5 comparing the microstruc-

TABLE III

SUMMARY OF ALLOYS, TREATMENTS AND MICROSTRUCTURAL OBSERVATIONS IN A STUDY TO ESTABLISH A HEAT TREATMENT TIME FOR TENTATIVE PHASE BOUNDARY LOCATIONS

| Alloy | Extrusion Charge | Intended Composition and (Chemical Analyses) Mg/Li | Li | Zn | Condition Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Third |
|---|---|---|---|---|---|---|---|---|---|---|
| L-42 | 179 | 15 | 6.1 (5.32) | 2.0 (2.04) | A.E. | - | - | 80 | 20 | - |
| | | | | | | 24 | 700 | 80 | 20 | - |
| | | | | | | 72 | 700 | 80 | 20 | - |
| L-49 | 183 | 17 | 5.6 | 4.0 | A.E. | - | - | 80 | 20 | - |
| | | | | | | 24 | 700 | 80 | 20 | - |
| | | | | | | 72 | 700 | 80 | 20 | - |
| L-50 | 184 | 17 | 5.5 (5.0) | 6.0 (5.6) | A.E. | - | - | 85 | 15 | - |
| | | | | | | 24 | 700 | 85 | 15 | - |
| | | | | | | 72 | 700 | 85 | 15 | - |
| L-84 | 218 | 30 | 3.2 | 2.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-85 | 219 | 30 | 3.1 | 4.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-162 | 290 | 10 | 8.0 (7.0) | 12.0 (12.1) | A.E. | - | - | 45 | 45 | 10 MgLiZn |
| | | | | | | 24 | 700 | 39 | 60 | 1 MgLiZn |
| | | | | | | 6 | 500 | 40 | 40 | 20 MgLiZn |
| | | | | | | 12 | 500 | 40 | 40 | 20 MgLiZn |
| | | | | | | 40 | 500 | 40 | 40 | 20 MgLiZn |
| | | | | | | 48 | 500 | 40 | 40 | 20 MgLiZn |
| | | | | | | 72 | 500 | 40 | 40 | 20 MgLiZn |

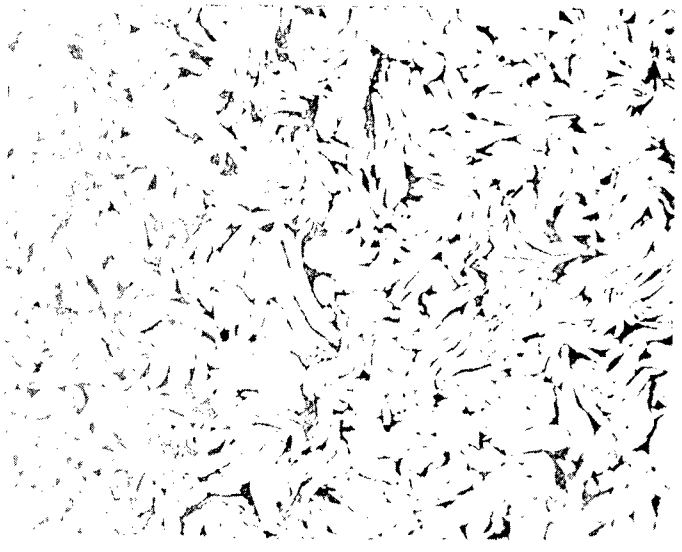


Figure 3

Neg: 251 Mag: 100X
Spec: M723 Etch: Salicylic
20 sec.

Traverse section of the center structure of asextruded alloy L-42 (charge 179, magnesium 5.32 lithium 2.04 zinc), showing approximately 80% alpha and 20% beta phases in a relatively uniform distribution.

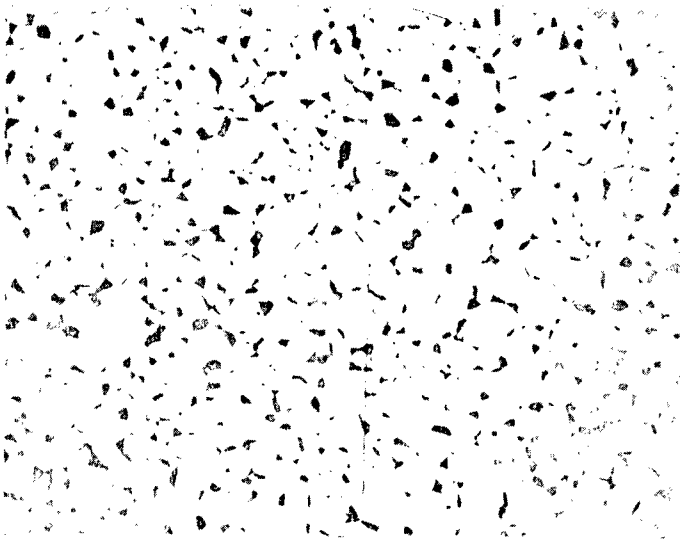


Figure 4

Neg: 253 Mag: 100X
Spec: M709 Etch: Salicylic
20 sec.

Typical appearance of the center structure of extruded alloy L-42 after heat treating 24 hours at 700°F (in a helium atmosphere in an aluminum container) and quenched in kerosene. The beta phase was agglomerated but there was little change in relative amounts of phases.

Figure 5

Neg: 255 Mag: 100X
Spec: M741 Etch: Salicylic
20 sec.

Same conditions as for Figure 4 but heat treated 72 hours. No significant change in the amount and distribution of phases was observed as a result of the increased time at temperature.

tures of specimens in the as-extruded and heat treated conditions. The only significant difference produced by the longer time at 700°F was to extend the depth of a surface layer depleted in the beta phase and to extend the condition which appears like an internal oxidation.

On this basis of these observations the heat treatment of 24 hours at 700°F was chosen for all alloys in both systems. This decision was considered justified because (a) the 24 hour treatment produced a sufficiently close approach to equilibrium for the purpose of the initial location of phase boundaries by the microscopic method, and, (b) the degree of surface attack of specimens and loss of lithium from the specimen increased as the heat treatment time was lengthened.

An investigation was also made to establish an acceptable time of heat treatment at 500°F for the initial study of phases present at this temperature. The intended composition of the alloy selected for this study was magnesium-8 lithium-12 zinc (L-162, charge 290). Alloy L-162 contained three phases in both the as-extruded condition and after 24 hours at 700°F heat treatment. These phases were alpha, beta and a phase tentatively identified as MgLiZn. Samples of alloy L-162 were sealed in Pyrex tubes in an atmosphere of helium, held 6, 12, 40, 48 and 72 hours at 500°F and quenched in kerosene. As shown in Table III microscopic examination indicated that all treatments produced a structure containing approximately 40% alpha, 40% beta and 20% MgLiZn phases. After heat treatment for 48 hours the microstructure had retained the

typically extruded stringer-like distribution of alpha and beta phases. The extruded structure was practically eliminated in the sample heat treated 72 hours and the microstructure was a uniform, equi-axed mixture of the three phases. There was no evidence of a significant degree of sample surface attack during the heat treatment at 500°F. From the results of this study, 72 hours was selected as a standard heat treatment time for magnesium-lithium-zinc and magnesium-lithium-aluminum alloys in the initial study of phases present at 500°F.

## Phase Boundaries in Magnesium-Lithium-Aluminum System

The microstructures of 57 alloys in this system were examined after heat treatment at 700°F and some of these alloys were examined after heat treatment at 500°F. In all cases the heat treated and as-extruded conditions of the same alloy were compared in order to follow the appearance or disappearance of a phase at elevated temperature. A summary is given in Table IV of the alloy intended compositions, chemical analyses when determined and estimation of phases present at 500°F and 700°F. Tentative locations of phase boundaries for the magnesium corner of this system at 700°F are shown in Fig. 6.

It is to be emphasized that the locations of phase boundaries in Fig. 6 are tentative, subject to additional investigation with new compositions and verification. The broken lines in Fig. 6 represent probable locations, estimated from evidence too limited in extent for more definite placement. The identification of the phase designated AlLi is tentative, awaiting the completion of an x-ray diffraction study of intermediate phases for verification. The anticipated extension of the phase, designated $Mg_{17}Al_{12}$ in the binary magnesium-lithium system, into the ternary system is indicated in Fig. 6 as a solid solution of magnesium, aluminum and lithium.

No phase relationships were plotted for the 500°F temperature level because the extent of investigation at this temperature had not provided a sufficient amount of data.

The only known literature on the constitution of ternary

TABLE IV

SUMMARY OF MAGNESIUM-LITHIUM-ALUMINUM ALLOY INTENDED COMPOSITIONS, CHEMICAL ANALYSES AND MICROSTRUCTURAL EVIDENCE OF PHASES PRESENT AT 500°F AND 700°F

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-19 | 153 | 19.0 | 5.0 | - | | | | | | |
| | | | (4.87) | - | A.E. | - | - | 100 | - | - |
| | | | | | | 72 | 500 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-20 | 151 | 11.5 | 8.0 | - | | | | | | |
| | | | (7.77) | - | A.E. | - | - | 50 | 50 | - |
| | | | | | | 72 | 500 | 50 | 50 | - |
| | | | | | | 72 | 700 | 50 | 50 | - |
| L-92 | 261 | 9.0 | 10.0 | - | | | | | | |
| | | | | | A.E. | - | - | - | 100 | - |
| | | | (14.9) | - | - | 24 | 700 | - | 100 | - |
| L-91 | 260 | 8.1 | 11.0 | - | | | | | | |
| | | | | | A.E. | - | - | 20 | 80 | - |
| | | | (10.5) | - | - | 24 | 700 | 10 | 90 | - |
| L-21 | 157 | 7.3 | 12.0 | - | | | | | | |
| | | | (11.85) | - | A.E. | - | - | | 100 | |
| | | | | | | 24 | 700 | | 100 | |
| L-43 | 175 | 5.7 | 15.0 | - | | | | | | |
| | | | (15.15) | - | A.E. | - | - | - | 100 | - |
| | | | | | | 72 | 500 | - | 100 | - |
| | | | | | | 96 | 700 | - | 100 | - |
| L-130 | 268 | 20.8 | 4.5 | 2.0 | | | | | | |
| | | | | | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| L-54 | 188 | 17.0 | 5.8 | 2.0 | | | | | | |
| | | | | | A.E. | - | - | 100 | - | - |
| | | | (5.4) | (1.9) | - | 24 | 700 | 100 | - | - |
| L-120 | 284 | 9.8 | 9.0 | 2.5 | | | | | | |
| | | | | | A.E. | - | - | 50 | 50 | - |
| | | | | | - | 24 | 700 | 50 | 50 | - |

TABLE IV, Cont'd.

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition: As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent: Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-121 | 258 | 8.3 | 10.5 (9.9-9.8) | 2.5 - | A.E. | - | - | 20 | 80 | - |
| | | | (9.7) | - | - | 24 | 700 | 15 | 85 | - |
| L-122 | 259 | 7.1 | 12.0 (11.6-11.9) | 2.5 - | A.E. | - | - | - | 100 | - |
| | | | (10.8) | - | - | 24 | 700 | - | 100 | - |
| L-131 | 269 | 31.3 | 3.0 | 3.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| L-52[a] | 189 | 17.0 | 5.6 | 4.0 | A.E. | - | - | 100 | - | - |
| | | | (4.9) | - | | 72 | 500 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-56[a] | 191 | 15.0 | 6.0 | 4.0 | | | | | | |
| | | | (4.7) | (3.87) | A.E. | - | - | 100 | - | - |
| | | | | | | 72 | 500 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-123 | 253 | 10.3 | 8.5 (8.0-7.9) | 4.0 (5.9) | A.E. | - | - | 40 | 40 | 20 AlLi |
| | | | (7.4) | (6.7) | - | 24 | 700 | 60 | 40 | - |
| L-142 | 279 | 7.73 | 11.0 | 4.0 | A.E. | - | - | 40 | 50 | 10 AlLi |
| | | | (10.3) | (6.0) | - | 24 | 700 | 25 | 75 | - |
| L-150 | 262 | 3.80 | 20.0 | 4.0 | A.E. | - | - | - | 100 | - |
| | | | | | | | 700 | - | 100 | - |
| L-26[a] | 158 | 10.0 | 8.5 | 5.0 | | | | | | |
| | | | (8.44) | (4.65) | A.E. | - | - | 40 | 40 | 20 AlLi |
| | | | | | | 72 | 500 | 40 | 40 | 20 AlLi |
| | | | | | | 72 | 700 | 50 | 50 | - |

(a) Notes at end of table.

TABLE IV, Cont'd.

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-22[a] | 154 | 8.0 | 10.6 | 5.0 | | | | | | |
| | | | (10.2) | (4.65) | A.E. | - | - | 10 | 87 | 5 AlLi |
| | | | | | | 96 | 700 | 5 | 95 | - |
| L-124 | 254 | 7.3 | 11.5 | 5.0 | | | | | | |
| | | | (10.0) | (5.6) | A.E. | - | - | 20 | 70 | 10 AlLi |
| | | | (10.3-10.3) | (5.3) | | 24 | 700 | 30 | 70 | - |
| L-58 | 193 | 6.0 | 13.6 | 5.0 | | | | | | |
| | | | (12.6) | (5.06) | A.E. | - | - | - | 100 | - |
| | | | | | | 96 | 700 | | 100 | - |
| L-74[b] | 207 | 99.0 | 0.9 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | 100 | - | - |
| | | | (0.8) | (5.0) | - | 24 | 700 | 100 | - | - |
| L-71[b] | 204 | 30.0 | 3.0 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | 100 | - | - |
| | | | (3.0) | (4.2) | - | 24 | 700 | 100 | - | - |
| L-53 | 192 | 17.0 | 5.5 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | - | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-57 | 196 | 15.0 | 5.9 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | 100 | - | - |
| | | | (5.2) | (5.0) | - | 24 | 700 | 100 | - | - |
| L-139 | 276 | 10.0 | 8.5 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | 55 | 35 | 10 AlLi |
| | | | (8.3) | (6.4) | - | 24 | 700 | 59 | 40 | 1 AlLi |
| L-128 | 255 | 6.5 | 12.5 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | - | 85 | 15 AlLi |
| | | | (11.0) | (4.8) | - | 24 | 700 | - | 100 | - |
| L-151 | 263 | 3.7 | 20.0 | 6.0 | | | | | | |
| | | | | | A.E. | - | - | - | 90 | 10 AlLi |
| | | | | | | 24 | 700 | - | 100 | - |

TABLE IV, Cont'd

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-148 | 265 | 5.2 | 15.0 | 7.0 | A.E. | - | - | - | 80 | 20 AlLi |
| | | | (14.2) | (6.1) | | 24 | 700 | - | 100 | - |
| L-140(b) | 277 | - | - | 7.5 | A.E. | - | - | 85 | - | 15 $Mg_{17}Al_{12}$ |
| | | | | (8.1) | - | 24 | 700 | 85 | - | 15 $Mg_{17}Al_{12}$ |
| L-141(b) | 278 | 91.0 | 1.0 | 8.0 | A.E. | - | - | 100 | - | - |
| | | | (0.9) | (6.3) | - | 24 | 700 | 100 | - | - |
| L-129 | 252 | 6.4 | 12.5 | 8.0 | A.E. | - | - | - | 80 | 20 AlLi |
| | | | (10.8) | (7.5) | - | - | 700 | 1 | 90 | 9 AlLi |
| L-152 | 264 | 3.6 | 20.0 | 8.0 | A.E. | - | - | - | 90 | 10 AlLi |
| | | | (19.2) | (8.2) | - | 24 | 700 | - | 100 | - |
| L-149 | 266 | 5.1 | 15.0 | 9.0 | A.E. | - | - | - | 75 | 25 AlLi |
| | | | (13.7) | (9.3) | - | 24 | 700 | - | 85 | 15 AlLi |
| L-135 | 273 | - | - | 10.0 | A.E. | - | - | 70 | - | 30 $Mg_{17}Al_{12}$ |
| | | | | | | 24 | 700 | 70 | - | 30 $Mg_{17}Al_{12}$ |
| L-136 | 274 | 29.0 | 3.0 | 10.0 | A.E. | - | - | 50 | 35 | 15 AlLi |
| | | | (9.0-8.8) | (9.8) | - | 24 | 700 | 50 | 35 | 15 AlLi |
| L-132 | 270 | 17.0 | 5.0 | 10.0 | A.E. | - | - | - | - | - |
| | | | (4.7) | (10.2) | - | 24 | 700 | 93 | - | 5 AlLi<br>2 $Mg_{17}Al_{12}$ |

TABLE IV, Cont'd

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-133 | 271 | 14.0 | 6.0 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | - | - | - |
| | | | (5.5) | (7.6) | - | 24 | 700 | 90 | - | 10 AlLi |
| L-134 | 272 | 11.9 | 7.0 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | - | - | - |
| | | | (6.7) | (8.2) | - | 24 | 700 | 90 | - | 10 AlLi |
| L-27 | 159 | 10.0 | 8.2 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | 45 | 20 | 35 AlLi |
| | | | | | | 72 | 500 | 50 | 10 | 40 AlLi |
| | | | | | | 72 | 700 | 45 | 20 | 35 AlLi |
| L-23 | 155 | 8.0 | 10.0 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | 33 | 33 | 33 AlLi |
| | | | | | | 72 | 500 | 35 | 35 | 30 AlLi |
| | | | | | | 72 | 700 | 35 | 35 | 30 AlLi |
| L-126 | 256 | 6.8 | 11.5 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | 15 | 70 | 15 AlLi |
| | | | (10.8) | (10.0) | - | 24 | 700 | 20 | 65 | 15 AlLi |
| L-59 | 194 | 6.0 | 12.9 | 10.0 | | | | | | |
| | | | | | A.E. | - | - | - | 60 | 40 AlLi |
| | | | | | | 72 | 500 | - | 65 | 35 AlLi |
| | | | | | | 72 | 700 | - | 65 | 35 AlLi |
| L-144(b) | 281 | 23.3 | 3.5 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | 80 | - | 20 $Mg_{17}Al_{12}$ |
| | | | (2.8) | (15.1) | - | 24 | 700 | 70 | - | 30 $Mg_{17}Al_{12}$ |
| L-143 | 280 | 16.0 | 5.0 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | - | - | - |
| | | | (4.8) | (15.4) | - | 24 | 700 | 80 | - | 5 AlLi, 15 $Mg_{17}Al_{12}$ |
| L-31 | 235 | 10.0 | 7.8 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | 90 | - | 10 AlLi |
| | | | (7.1) | (12.5) | - | 24 | 700 | 90 | - | 10 AlLi |

TABLE IV, Cont'd

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-24 | 156 | 8.0 | 9.4 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | 40 | 25 | 35 AlLi |
| | | | | | | 72 | 500 | 40 | 25 | 35 AlLi |
| | | | | | | 72 | 700 | 40 | 25 | 35 AlLi |
| L-127 | 257 | 6.7 | 11.0 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | 30 | 35 | 35 AlLi |
| | | | | | | 24 | 700 | 38 | 38 | 24 AlLi |
| L-60 | 195 | 6.0 | 12.2 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | 30 | 50 | 20 AlLi |
| | | | | | | | 500 | 25 | 50 | 25 AlLi |
| | | | | | | 72 | 700 | 40 | 30 | 30 AlLi |
| L-153 | 287 | 4.7 | 15.0 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | - | 70 | 30 AlLi |
| | | | (13.9) | (15.8) | - | 24 | 700 | - | 70 | 30 AlLi |
| L-155 | 285 | 3.3 | 20.0 | 15.0 | | | | | | |
| | | | | | A.E. | - | - | - | 90 | 10 AlLi |
| | | | (18.9) | (16.0) | - | 24 | 700 | - | 90 | 10 AlLi |
| L-146 | 283 | 21.9 | 3.5 | 20.0 | | | | | | |
| | | | | | A.E. | - | - | 50 | - | 50 $Mg_{17}Al_{12}$ |
| | | | | | | 24 | 700 | 50 | - | 50 $Mg_{17}Al_{12}$ |
| L-145 | 282 | 15.0 | 5.0 | 20.0 | | | | | | |
| | | | | | A.E. | - | - | 60 | - | 40 $Mg_{17}Al_{12}$ |
| | | | (4.6) | (23.3) | - | 24 | 700 | 70 | - | 30 $Mg_{17}Al_{12}$ |
| L-32 | 233 | 10.0 | 7.3 | 20.0 | | | | | | |
| | | | | | A.E. | - | - | - | - | - |
| | | | (6.8) | (17.2) | - | 24 | 700 | 90 | - | 10 AlLi |
| L-25[a] | 161 | 8.0 | 8.9 | 20.0 | | | | | | |
| | | | (8.54) | (18.9) | A.E. | - | - | 80 | - | 20 AlLi |
| | | | | | | | 500 | 80 | - | 20 AlLi |
| | | | | | | | 700 | 80 | - | 20 AlLi |

TABLE IV, Cont'd

| Alloy | Extrusion Charge | Intended Composition Mg/Li | Li | Al | Condition: As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-61 | 234 | 6.0 | 11.4 | 20.0 | A.E. | - | - | 33 | 33 | 33 AlLi |
| | | | (9.2) | (20.3) | - | 24 | 700 | 40 | 40 | 20 AlLi |
| L-154 | 286 | 4.3 | 15.0 | 20.0 | A.E. | - | - | - | 60 | 40 AlLi |
| | | | (12.9) | (21.3) | - | 24 | 700 | 5 | 60 | 35 AlLi |

Notes:

(a) The estimation of phases present for the alloy in this tabulation is different from results reported in Monthly Progress Reports. Re-examination of these alloys after more experience was gained in metallographic specimen preparation indicated a consistent tendency to over-estimate the relative amount of the phase believed to be AlLi.

(b) The samples for the alloy were protected by "Keepbryte" during heat treatment and were not sealed in Pyrex tubes.

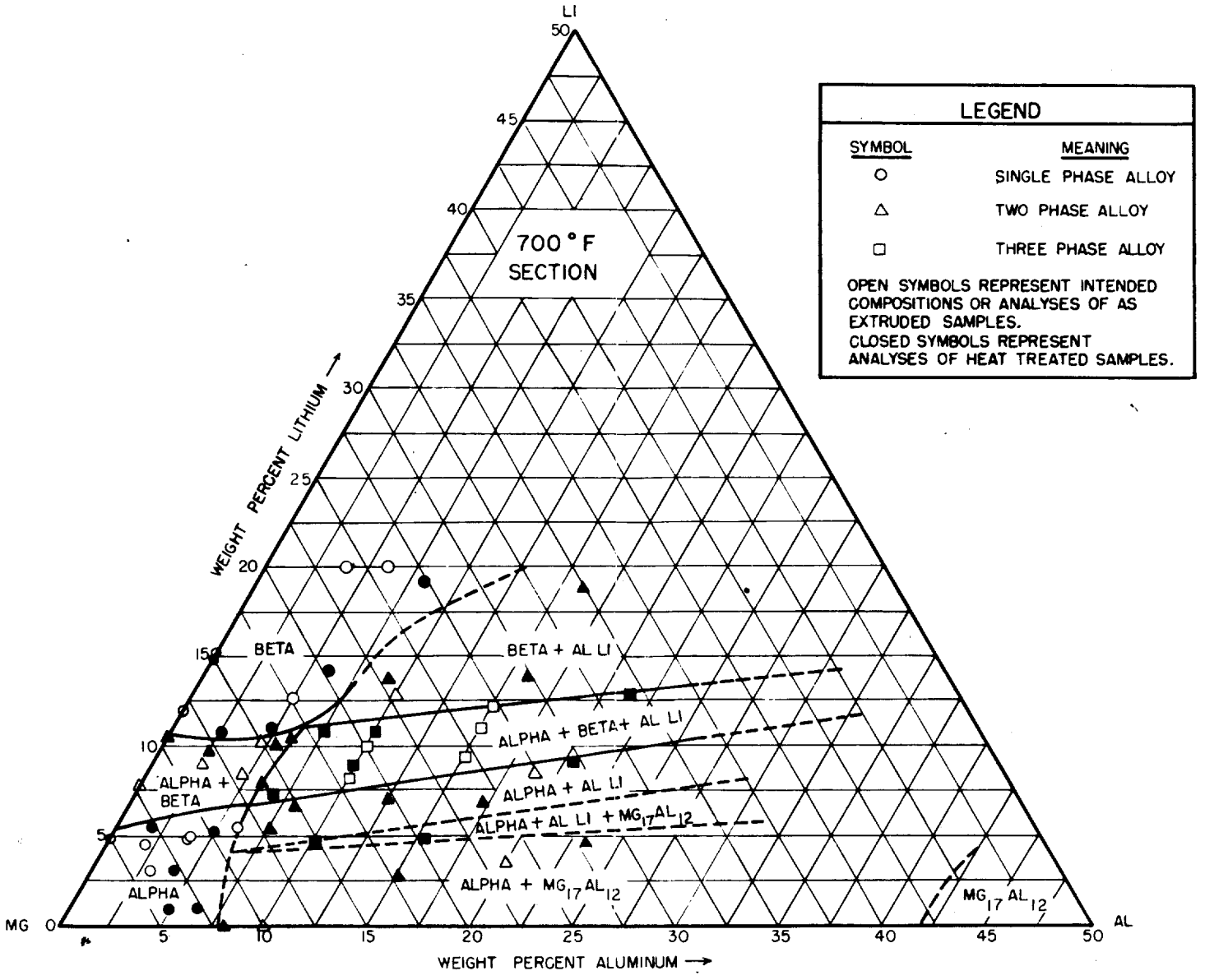



FIGURE 6

APPROXIMATE LOCATIONS OF PHASE BOUNDARIES IN THE MAGNESIUM CORNER OF THE MAGNESIUM-LITHIUM-ALUMINUM SYSTEM AT 700°F. THE IDENTIFICATION OF THE PHASES DESIGNATED AL LI AND $MG_{17}AL_{12}$ IS CONSIDERED TENTATIVE.

magnesium-lithium-aluminum alloys is the Russian work of Shamrai[2]. In order to compare more directly with the present work, Shamrai's 752°F (400°C) section was replotted in terms of weight percentage of elements and the results are shown in Fig. 7. Since the descriptive portion of the Russian work was not available for translation into English the intended accuracy of the results is not known and the exact significance of the broken-line phase boundaries is unknown. The legend of the phase identification, using Roman numerals as given by Shamrai, was derived from consideration of the positions of phases.

In comparing the results presented in Figs. 6 and 7, it is emphasized that the present work is considered tentative. In addition, the 52°F temperature difference in sections plotted should be noted. Up to this time, our experimental work has not recognized the phases designated by Shamrai as $AlLi_2$ and $MgAl_2Li$. As noted in the x-ray diffraction section of this report a study is in progress to establish a means for identifying these and other phases possibly encountered in the system. A significant difference between the two plots is in the extent of the beta and alpha plus beta phase fields. Possibly a portion of this difference could be attributed to the difference in temperature of the respective sections through the ternary systems. A more specific comparison is not considered justified on the basis of the information at hand.

An examination of alloys for the 500°F section is in progress. New compositions have been selected for preparation

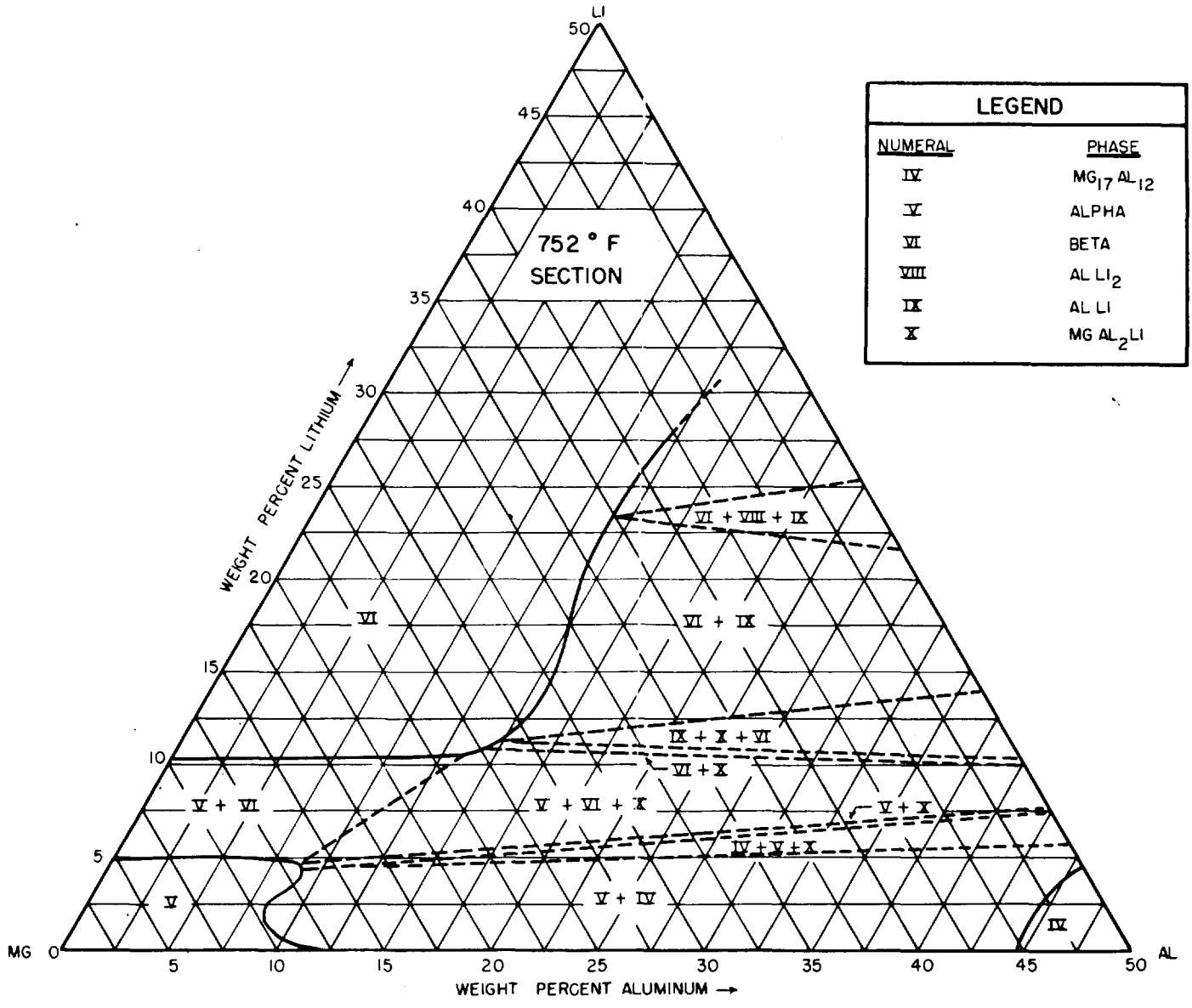



FIGURE 7

LOCATIONS OF PHASE BOUNDARIES IN THE MAGNESIUM CORNER OF THE MAGNESIUM-LITHIUM-ALUMINUM SYSTEM AT 752°F (400°C) AS REPLOTTED FROM SHAMRAI'S TERNARY DIAGRAM.[2] THE LEGEND GIVES SHAMRAI'S PHASE NOMENCLATURE.

and evaluation on the basis of the microscopic evidence at 700°F. These compositions have been chosen to assist locating the boundaries between the following phase fields:

beta and beta plus AlLi

alpha and alpha plus $Mg_{17}Al_{12}$

alpha plus $Mg_{17}Al_{12}$ and alpha plus AlLi plus $Mg_{17}Al_{12}$

alpha plus AlLi and alpha plus AlLi plus $Mg_{17}Al_{12}$.

In addition, compositions have been selected to extend the study to higher lithium and aluminum concentrations.

Phase Boundaries in the Magnesium-Lithium-Zinc System

The microscopic examination of alloys in this system was conducted in a manner similar to the examination of magnesium-lithium-aluminum alloys and simultaneously with that evaluation. The microstructures of ternary alloys in this system examined up to this time contained one or more of three phases - alpha, beta and an intermediate phase believed to be MgLiZn. The identity of this intermediate phase is considered tentative, awaiting the completion of an x-ray diffraction study of intermediate phases for verification. The phase MgZn was observed in a binary magnesium-6 zinc alloy but its presence was not recognized in the ternary alloys studied up to this time. A summary of the results of microscopic determination of phases present in 48 alloys at the 700°F temperature level is given in Table V. Tentative locations of phase boundaries for the magnesium corner of this system at 700°F are shown in Fig. 8.

As noted for the magnesium-lithium-aluminum system, the phase boundary locations in Fig. 8 are considered tentative. None of the alloys in this system prepared and examined at this stage of the study revealed a sufficient amount of the phase MgZn to aid in establishing a metallographic identification procedure.

Microscopic examination of alloys for the 500°F section was started but the results obtained were considered too limited for reporting at this time.

As can be seen from Table V some of the alloys in the as-extruded condition revealed a significant extent of an inter-

TABLE V

SUMMARY OF MAGNESIUM-LITHIUM-ZINC ALLOY INTENDED COMPOSITIONS, CHEMICAL ANALYSES AND MICROSTRUCTURAL EVIDENCE OF PHASES PRESENT AT 700°F

| Alloy | Extrusion Charge | Intended Composition and (Chemical Analyses) Mg/Li | Li | Zn | Condition: As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent: Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-78 | 211 | - | - | 2.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 29 | 700 | 100 | - | - |
| L-79 | 212 | - | - | 4.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 29 | 700 | 100 | - | - |
| L-80 | 213 | - | - | 6.0 | A.E. | - | - | 90 | - | 10 MgZn |
| | | | | | | 29 | 700 | 100 | - | - |
| L-81 | 214 | 99 | 1.0 | 2.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 29 | 700 | 100 | - | - |
| L-82 | 215 | 99 | 1.0 | 4.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 29 | 700 | 100 | - | - |
| L-83 | 216 | 99 | 0.9 | 6.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 29 | 700 | 100 | - | - |
| L-84 | 218 | 30 | 3.2 | 2.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-85 | 219 | 30 | 3.1 | 4.0 | A.E. | - | - | 100 | - | - |
| | | | | | | 24 | 700 | 100 | - | - |
| | | | | | | 72 | 700 | 100 | - | - |
| L-119 | 249 | 30 | 3.0 | 8.0 | A.E. | - | - | 70 | - | 30 MgZn or MgLiZn[a] |
| | | | (2.5) | (8.5) | - | 24 | 700 | 100 | - | - |
| L-164 | 292 | 30 | 3.0 | 8.0 | A.E. | - | - | 90 | - | 10 MgLiZn |
| | | | (2.5) | (7.8) | - | 24 | 700 | 90 | - | 10 MgLiZn |
| L-116 | 246 | 30 | 2.9 | 10.0 | A.E. | - | - | 50 | - | 50 MgZn |
| | | | (2.3) | (10.0) | - | 24 | 700 | 85 | - | 15 Melted[b] |

a. Notes at end of table.

TABLE V, Cont'd

| Alloy | Extrusion Charge | Intended Composition and (Chemical Analyses) Mg/Li | Li | Zn | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-117 | 247 | 30 | 2.7 | 15.0 | A.E. | - | - | 60 | - | 40 MgZn or MgLiZn[a] |
| | | | (2.2) | (15.0) | - | 24 | 700 | 80 | - | 20 MgLiZn |
| L-118 | 248 | 30 | 2.6 | 20.0 | A.E. | - | - | 50 | - | 50 MgLiZn |
| | | | (2.1) | (21.9) | - | 24 | 700 | 60 | - | 40 MgLiZn |
| L-48 | 182 | 17 | 5.8 | 2.0 | A.E. | - | - | 100 | - | - |
| | | | (4.7) | (1.6) | - | 29 | 700 | 100 | - | - |
| L-49 | 183 | 17 | 5.6 | 4.0 | A.E. | - | - | 80 | 20 | - |
| | | | | | | 24 | 700 | 80 | 20 | d |
| | | | | | | 72 | 700 | 80 | 20 | d |
| L-50 | 184 | 17 | 5.5 | 6.0 | A.E. | - | - | 85 | 15 | - |
| | | | (5.0) | (5.6) | - | 24 | 700 | 85 | 15 | d |
| | | | | | | 72 | 700 | 85 | 15 | d |
| L-42 | 179 | 15 | 6.1 | 2.0 | - | - | - | 80 | 20 | - |
| | | | (5.32) | (2.04) | A.E. | 24 | 700 | 80 | 20 | - |
| | | | | | | 72 | 700 | 80 | 20 | - |
| L-45 | 180 | 15 | 6.0 | 4.0 | A.E. | - | - | 85 | 15 | d |
| | | | (5.5) | (3.6) | - | 24 | 700 | 85 | 15 | d |
| L-46 | 181 | 15 | 5.9 | 6.0 | A.E. | - | - | 75 | 25 | d |
| | | | | | | 24 | 700 | 75 | 25 | d |
| L-111 | 245 | 15 | 5.8 | 8.0 | A.E. | - | - | 85 | 10 | 5 MgLiZn |
| | | | (5.2) | (3.6) | - | 24 | 700 | 90 | 10 | - |
| L-112 | 250 | 15 | 5.6 | 10.0 | A.E. | - | - | 80 | - | 20 MgLiZn |
| | | | (4.8) | (9.2) | - | 24 | 700 | 85 | 15 | - |
| L-113 | 267 | 15 | 5.3 | 15.0 | - | - | - | - | - | - |
| | | | (4.6) | (14.7) | - | 24 | 700 | 55 | 30 | 15 MgLiZn |
| L-114 | 251 | 15 | 5.0 | 20.0 | A.E. | - | - | 50 | - | 50 MgLiZn |
| | | | (3.9) | (20.5) | - | 24 | 700 | 70 | - | 30 MgLiZn |
| L-33 | 174 | 10 | 8.9 | 2.0 | A.E. | - | - | 40 | 60 | - |
| | | | | | | 24 | 700 | 40 | 60 | - |

TABLE V, Cont'd

| Alloy | Extrusion Charge | Intended Composition and (Chemical Analyses) Mg/Li | Li | Zn | Condition As Ext'd. | Heat Treatment Time (Hr) | Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-34 | 173 | 10 | 8.7 | 4.0 | A.E. | - | - | 30 | 70 | - |
| | | | | | | 24 | 700 | 30 | 70 | - |
| L-35 | 172 | 10 | 8.5 | 6.0 | A.E. | - | - | 45 | 55 | - |
| | 237 | | | | | 29 | 700 | 40 | 60 | - |
| L-36 | 171 | 10 | 8.4 | 8.0 | - | - | - | - | - | - |
| | 166 | | (8.2) | (7.96) | A.E. | - | - | 30 | 65 | 5 MgLiZn |
| | | | | | | 29 | 700 | 30 | 70 | - |
| L-161 | 289 | 10 | 8.2 | 10.0 | A.E. | - | - | 40 | 60 | - |
| | | | (7.4) | (10.5) | - | 24 | 700 | 40 | 60 | - |
| L-162 | 290 | 10 | 8.0 | 12.0 | A.E. | - | - | 45 | 45 | 10 MgLiZn |
| | | | (7.0) | (12.1) | - | 24 | 700 | 39 | 60 | 5 MgLiZn |
| L-109 | 244 | 10 | 7.8 | 15.0 | - | - | - | - | - | - |
| | | | (16.1) | - | A.E. | - | - | - | 18 | 80 ppt. + 5 MgLiZn[c] |
| | | | (15.8) | (18.2) | - | 24 | 700 | - | 35 | 65 ppt.[c] |
| | | | | | | 24 | 700 | - | 60 | 40 ppt.[c] |
| L-163 | 291 | 10 | 7.3 | 20.0 | A.E. | - | - | 35 | 35 | 30 MgLiZn |
| | | | | | | 24 | 700 | 35 | 45 | 20 MgLiZn |
| L-37 | 170 | 8 | 10.9 | 2.0 | - | - | - | - | - | - |
| | | | (10.9) | (2.03) | A.E. | - | - | - | 100 | - |
| | | | (10.2) | - | - | 29 | 700 | - | 100 | - |
| L-38 | 169 | 8 | 10.7 | 4.0 | - | - | - | - | - | - |
| | | | (10.65) | - | A.E. | - | - | - | 100 | - |
| | | | (9.9) | - | - | 24 | 700 | - | 100 | - |
| L-40 | 167 | 8 | 10.2 | 8.0 | A.E. | - | - | 90 | - | 10 MgLiZn |
| | | | (8.7) | - | - | 29 | 700 | 100 | - | - |
| L-158 | 293 | 8 | 9.8 | 12.0 | - | - | - | - | - | - |
| | | | | (11.7) | A.E. | - | - | 10 | 85 | 5 MgLiZn |
| | | | (8.4) | (12.8) | - | 24 | 700 | 10 | 90 | - |
| L-159 | 294 | 8 | 9.5 | 15.0 | A.E. | - | - | - | 90 | 10 MgLiZn |
| | | | (9.6) | (11.2) | - | 24 | 700 | - | 100 | - |

TABLE V, Cont'd

| Alloy | Extrusion Charge | Intended Composition and (Chemical Analyses) Mg/Li | Li | Zn | Condition As Ext'd. | Heat Treatment Time (Hr) | Heat Treatment Temp. (°F) | Phases Present and Estimation of Relative Amounts Percent Alpha | Beta | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| L-160 | 288 | 8 | 8.9 | 20.0 | A.E. | - | - | - | 70 | 20 MgLiZn, 10 ppt.[c] |
| | | | (7.0) | (20.0) | - | 24 | 700 | 3 | 85 | 12 MgLiZn |
| L-96 | 238 | 7 | 12.3 | 2.0 | A.E. | - | - | - | 100 | - |
| | | | (10.5) | (1.8) | - | 24 | 700 | - | 100 | - |
| L-97 | 239 | 7 | 12.0 | 4.0 | A.E. | - | - | - | 100 | - |
| | | | (10.9) | - | - | 24 | 700 | - | 100 | - |
| L-98 | 240 | 7 | 11.7 | 6.0 | A.E. | - | - | - | 90 | 10 MgLiZn |
| | | | | | | 24 | 700 | - | 100 | - |
| L-99 | 241 | 7 | 11.5 | 8.0 | A.E. | - | - | - | 90 | 10 MgLiZn |
| | | | (9.6) | (7.4) | - | 24 | 700 | - | 100 | - |
| L-100 | 242 | 7 | 11.2 | 10.0 | A.E. | - | - | - | 80 | 20 MgLiZn |
| | | | (9.8) | (9.0) | - | 24 | 700 | - | 100 | - |
| L-102 | 243 | 7 | 10.0 | 20.0 | A.E. | - | - | 50 | 20 | 30 MgLiZn |
| | | | (6.0) | (20.4) | - | 24 | 700 | 45 | 45 | 10 MgLiZn |
| L-87 | 220 | 6 | 14.0 | 2.2 | A.E. | - | - | - | 100 | - |
| | | | | | | 29 | 700 | - | 100 | - |
| L-88 | 221 | 6 | 12.9 | 10.0 | A.E. | - | - | - | 100 | - |
| | | | | | | 24 | 700 | - | 100 | - |
| L-89 | 222 | 6 | 12.2 | 15.0 | A.E. | - | - | - | 100 | - |
| | | | | | | 24 | 700 | - | 100 | - |
| L-90 | 223 | 6 | 11.4 | 20.0 | A.E. | - | - | - | 99 | 5 MgLiZn |
| | | | | | | 24 | 700 | - | 80 | 10 MgLiZn |
| | | | | | | | | | | 10 ppt.[c] |
| | | | (9.9) | (20.2) | - | 24 | 700 | - | 90 | 10 MgLiZn |

a. Secondary phase finely divided and not clearly resolved.
b. Specimen showed evidence of grain boundary and eutectic melting.
c. Specimen contained a precipitate similar to that shown in Fig. 11. The precipitate was found only at grain boundaries, and was believed to have formed during quenching from the heat treating temperature (or during cooling from the temperature of extrusion in the as-extruded specimen).
d. Third phase may have been present.

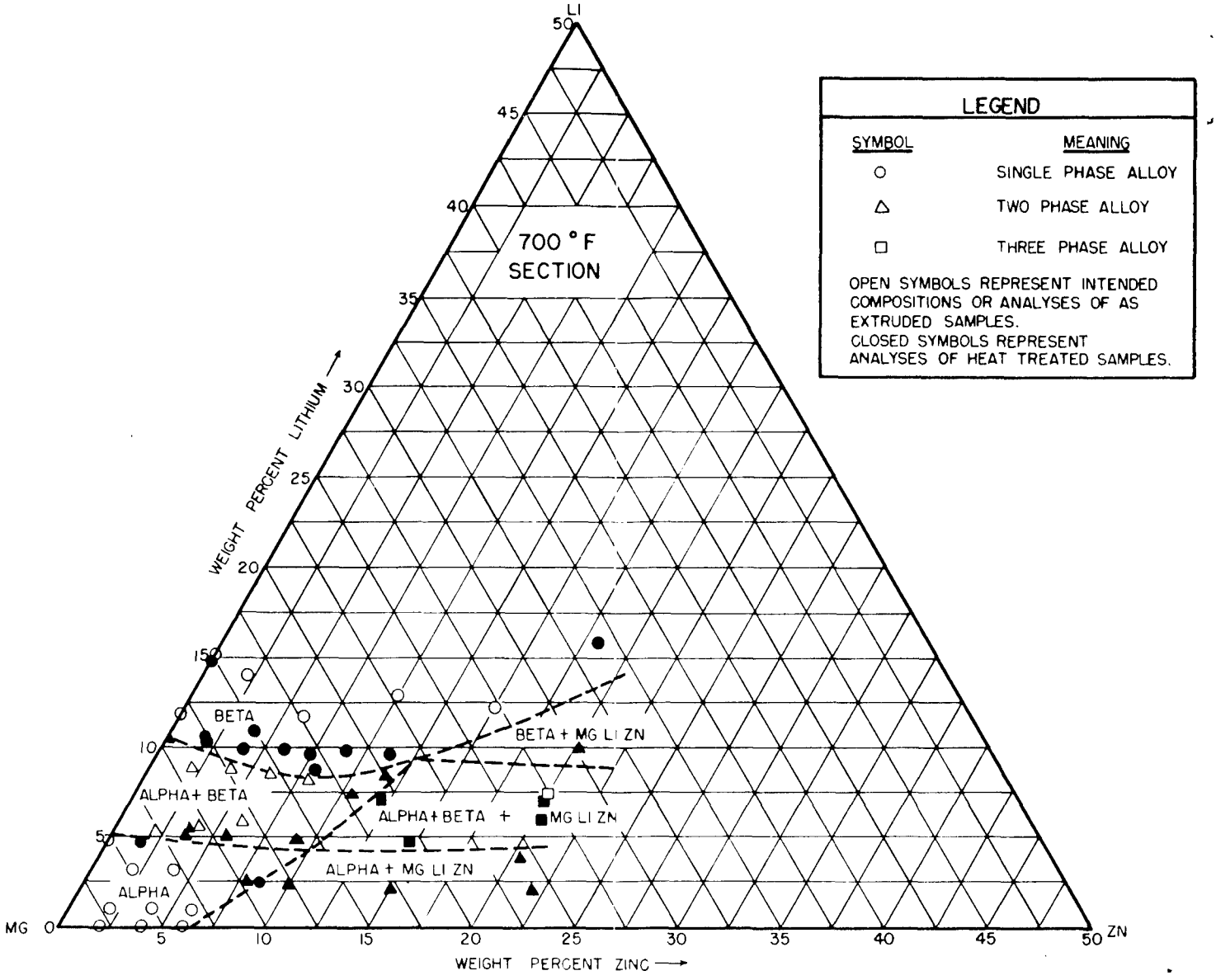



FIGURE8

APPROXIMATE LOCATIONS OF PHASE BOUNDARIES IN THE MAGNESIUM CORNER OF THE MAGNESIUM-LITHIUM-ZINC SYSTEM AT 700°F. THE IDENTIFICATION OF THE PHASE DESIGNATED MG LI ZN IS CONSIDERED TENTATIVE.

mediate phase believed to be MgLiZn. After heat treatment of these alloys at 700°F, the phase had disappeared. This condition is illustrated in Figs. 9 and 10 for alloy L-40. In the as-extruded condition, Fig. 9, approximately 10% of an intermediate phase, probably MgLiZn, was present. After 24 hours at 700°F, Fig. 10, no distinct evidence of this phase was detected and the structure appeared to be a complete beta solid solution. The approximate order of grain growth in 24 hours at 700°F for the single phase beta alloys is also illustrated in Fig. 10.

A feathery grain boundary precipitation was observed in three alloys examined. These alloys contained the order of 20% zinc and had the following chemical analyses:

L-90 magnesium-9.9 lithium-20.2 zinc

L-109 magnesium-15.8 lithium-18.2 zinc

L-160 magnesium-7.0 lithium-20.0 zinc

In the as-extruded condition alloy L-90 was found to be practically a complete beta solid solution with less than 5% of an intermediate phase, considered to be MgLiZn. No distinct evidence of a precipitation from solid solution was observed. After heat treating the alloy 24 hours at 700°F and quenching in kerosene it was estimated that the structure contained 80% beta 10% MgLiZn and 10% of a grain boundary precipitate. This condition is illustrated in Fig. 11. A duplicate specimen was heat treated and quenched using the same conditions. Microscopic examination indicated that the structure was 90% beta and 10% MgLiZn phases, with no distinct evidence of grain boundary precipitation. It is

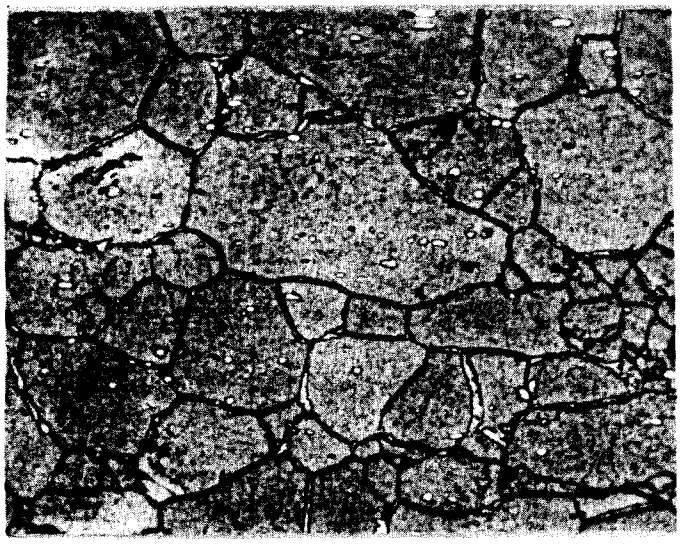


Figure 9

Neg: 237 Mag: 500X
Spec: M722 Etch: Picral-Acetic, 5 sec.

Longitudinal section of as-extruded alloy L-40 (charge 167, magnesium-10.2 lithium-8 zinc). The structure was estimated to be 90% beta phase and 10% intermediate phase (believed to be MgLiZn) located within grains and along grain boundaries as discrete particles.

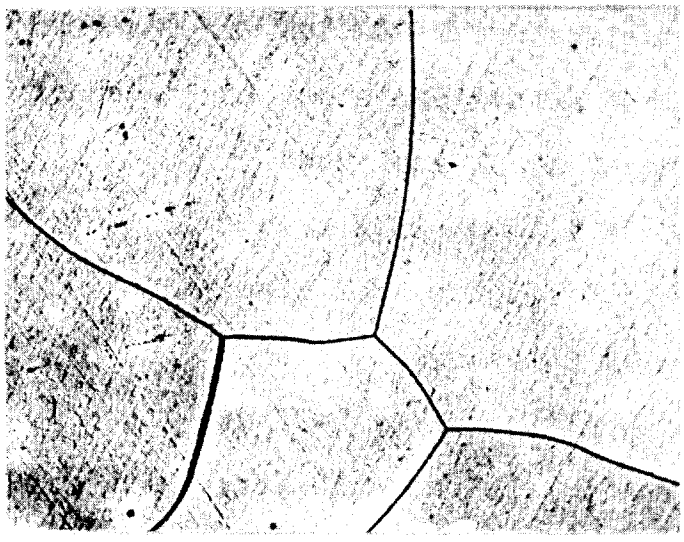


Figure 10

Neg: 238 Mag: 500X
Spec: M784 Etch: Picral-Acetic, 5 sec.

Longitudinal section from extruded alloy L-40 after heat treating 24 hours at 700°F in a helium atmosphere in an aluminum container and quenched in kerosene. The structure was essentially a complete beta solid solution. Note the grain coarsening.

Figure 11

Neg: 241 Mag: 2000X
Spec: M718 Etch: Salicylic 5 sec.

Longitudinal section from extruded alloy L-90 (charge 223, magnesium-11.4% lithium-20% zinc) after heat treating 24 hours at 700°F in a helium atmosphere in an aluminum container and quenched in kerosene. The structure was estimated to be 10% primary MgLiZn and 10% of a precipitate phase (believed to be MgLiZn) in a beta matrix.

believed that the original sample of L-90 was quenched too slowly to retain the solid solution structure.

Alloy L-160 in the as-extruded condition was estimated to contain 70% beta, 20% MgLiZn and 10% of a grain boundary precipitation. After heating the microstructure was estimated to be approximately 5% alpha, 85% beta and 10% MgLiZn with no evidence of grain boundary precipitation. In the as-extruded condition, alloy L-109 had a microstructure of 18% beta, less than 5% MgLiZn and approximately 80% feathery grain boundary precipitate. Heat treatment at 700°F and quenching in kerosene produced a microstructure of 35% beta and 65% of the precipitated condition. The heat treatment at 700°F was repeated and the sample was quenched as rapidly as possible in cold kerosene. Examination indicated that the extent of the precipitated condition was reduced to approximately 40% of the structure and the remainder was beta phase. These observations indicated that alloy L-109 was a beta solid solution at 700°F but that quenching in cold kerosene did not prevent precipitation. Work was continued to develop a method for more rapid quenching in an effort to substantiate this indication.

Alloys L-37 and L-38 were reported previously to contain small percentages of the alpha phase after heat treatment at 700°F. Upon re-examination of these alloys it was found that the alloys were completely beta solid solution. This difference in observation was due to a misinterpretation of the etched microstructure. In the original examination, standard acetic picral (see Appendix IV) was used. This solution produced a heavy, light

to dark brown stain on the structure. Discontinuities in the film, particularly along grain boundaries, revealed a light colored phase, originally believed to be alpha phase. Repeated examination of these two and other alloys with a variety of etchants indicated that the alpha phase was not present.

The examination of alloys for the 500°F section in this system is in progress. New compositions have been selected and are being prepared for evaluation at 700°F and 500°F. The need for a more thorough study in the general region of 0-10% lithium and 7.5-20. zinc was indicated by the results of the metallographic survey at 700°F. This region is believed to contain all phase fields of interest in this system.

Status of Metallographic Study and Plans for Future Work

The initial results of microscopic examination at 700°F in both systems under investigation have indicated the need for additional study for more precise location of phase boundaries at this temperature. New compositions have been selected and are being prepared for evaluation.

The microscopic examination of specimens for the 500°F section is in progress. This work will be completed before starting the study of additional sections in the ternary systems. The new sections tentatively selected are 150°F, 200°F and 300°F.

A correlation between the x-ray diffraction identification of intermediate phases in the two systems and the identification of phases in the microstructure is in progress. Until this work is completed the identity of intermediate phases in the two

systems must be considered tentative.

Although the method of sealing specimens within glass tubing in a helium atmosphere was effective in sample protection during heat treatment, efforts will be continued to apply the aluminum container method for routine heat treatment. This is considered advisable to accelerate the heat treatment program.

The investigation of a procedure of careful abrading and cleaning the entire surface of specimens prior to heat treatment is planned to determine its effect on the occurrence of the general condition of surface attack of specimens during the heat treatment.

# ELECTRICAL RESISTIVITY STUDY OF MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

## Purpose of the Equipment

Equipment was designed, constructed and assembled to adapt the technique of electrical resistivity determinations for establishing phase boundaries in magnesium-lithium base ternary alloys. Since the apparatus was designed to make the determinations at an elevated temperature of interest, it was anticipated that the method would be of considerable additional help in predicting the length of time required to reach equilibrium for specimens quenched for microscopic study. This equilibrium condition would be recognized when resistance values became constant at a given temperature.

## Development of Equipment

The initial design, construction and testing of the electrical resistivity apparatus was described in the Summary Report AF-TR-6174(1). The essential components of the equipment were:

1. Electrical resistance furnace and temperature control circuit for heating specimens.
2. Specimen container, a massive block of commercially pure aluminum to totally enclose specimens. This container was intended to serve the dual function of retaining a protective atmosphere around specimens and for minimizing temperature fluctuations.
3. Specimen mounting block and specimen holders enclosed in the container, with provision for mounting six speci-

mens at one time.

4. A direct current circuit for establishing and maintaining a constant value of current through the specimens.
5. A potentiometric circuit for measuring the individual potential drops across a 2 in. length of specimen and across a reference standard resistance of 0.001 ohms.

Preliminary operation of the assembled apparatus indicated that all parts performed satisfactorily except the specimen container. During operation, the rate of loss of the protective atmosphere from the container was considered excessive. The entire design of the equipment was re-considered and the following modifications were incorporated in a new assembly:

1. A new specimen container cover was made to provide for the removal of all electrical circuit wiring and entry of a protective atmosphere through a single tube.
2. The specimen container was fitted with a new sealing rim to accomodate the new container cover and to provide eight stainless steel studs for sealing.
3. A copper-asbestos sandwich-type gasket was selected as the most appropriate means of providing an effective seal for the specimen container cover.
4. A new system of hermetic seals was constructed for all electrical connections. The electrical connections were led through a steel pipe to a position sufficiently removed from the container cover to permit the use of soldered wire terminals.

5. Specimen holders were re-designed to provide greater ease of inserting and removing specimens and an improved system of current and potential connections.

Details of construction of these modifications are discussed in Appendix V. A photograph of the modified container, container cover, electrical terminal head assembly and specimen mounting system is shown in Fig. 12. The entire apparatus for electrical resistivity determinations is illustrated in Fig. 13.

Preliminary Operation of Equipment

The assembled electrical resistivity apparatus was operated under a number of different conditions of protective atmospheres in an effort to establish a procedure for consistently satisfactory performance.

The first trial runs of the unit were made under conditions of a positive pressure of helium being maintained during the test period. The tank helium was first passed through a purification train before entering the specimen container. The train consisted of (1) a mercury seal safety valve on the input end of the train leading from the tank pressure regulator, (2) a Milligan bottle containing concentrated sulfuric acid, (3) a tube furnace containing calcium chips at 1200°F, (4) a second tube furnace containing lithium foil at 320°F and, (5) a mercury monometer to measure pressure at the container input. It was believed that this system would eliminate any harmful amounts of $O_2$ or $H_2O$ in the gas.

Samples of 1/8 in. diameter extruded magnesium-lithium-

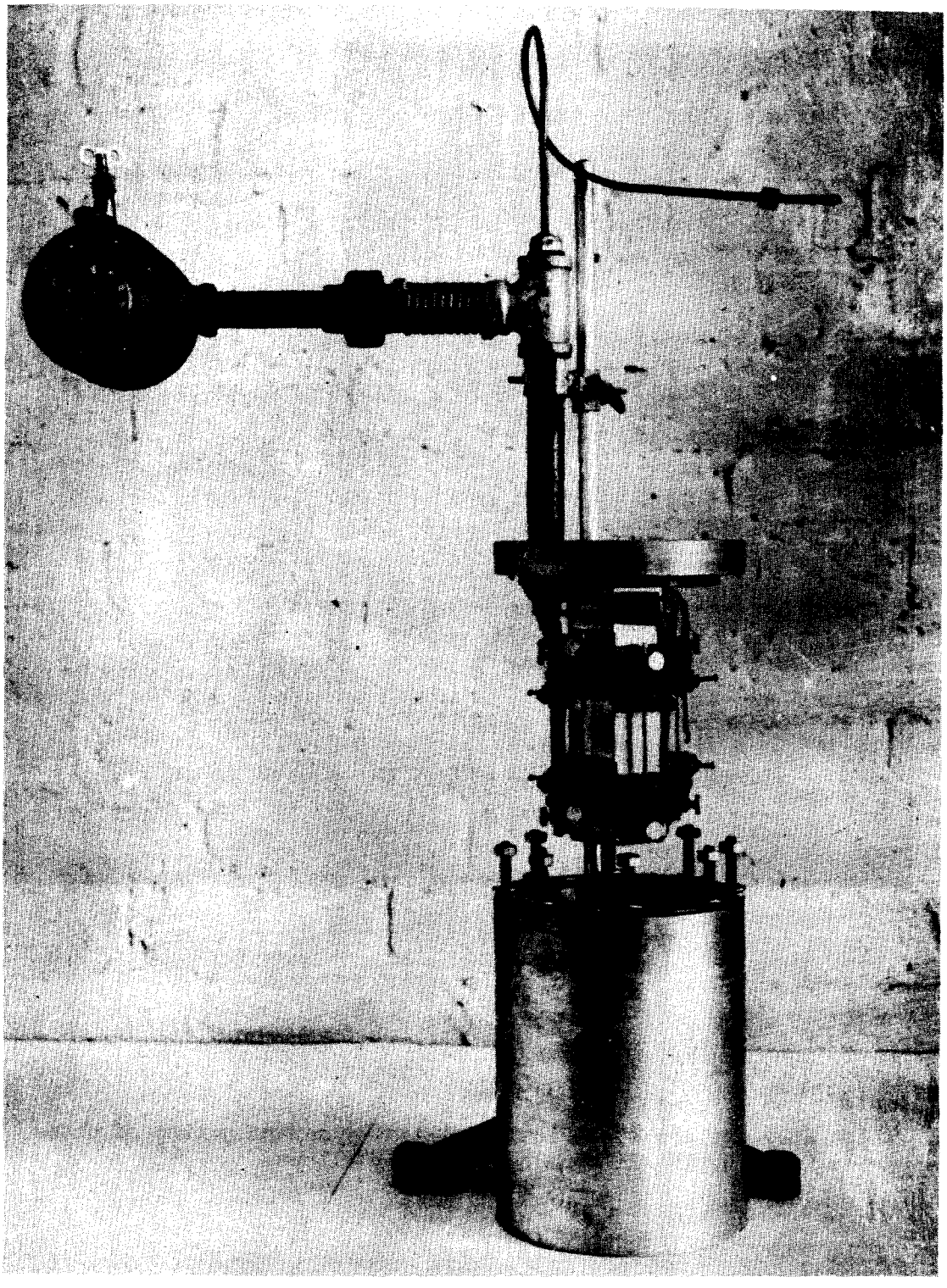


Figure 12

Modified specimen container, container cover, electrical terminal head assembly and specimen mounting system for electrical resistivity apparatus.

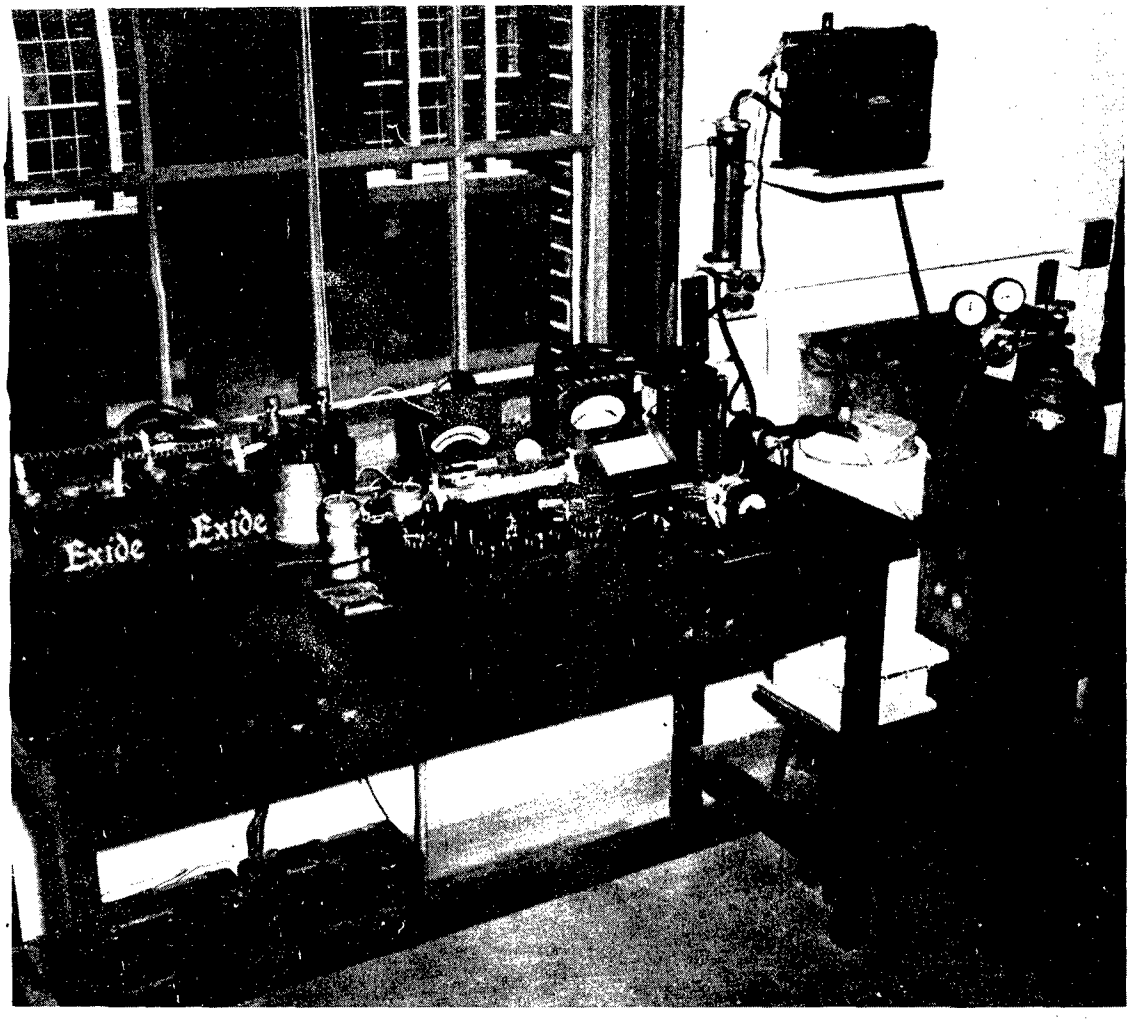



Figure 13

Apparatus for the electrical resistivity determination of phase boundaries in magnesium-lithium base ternary alloys.

aluminum ternary alloys were selected and placed in the container. The specimen ends were filed bright to provide good contact and the circuit operation was checked before final assembly. Container gaskets were cut from 1/8 in. Durabla[a] sheet, a commercial asbestos graphite gasket material. Furnace temperature was controlled with a Tagliabue photo-electric controller and a thermocouple in the furnace windings. Internal container temperature was measured with a Brown Potentiometer and a thermocouple placed inside the container adjacent to the mounted specimens.

Before starting a run, the container was pumped down with a Cenco "Hyvac" pump and thoroughly flushed with several cubic feet of helium. The final setting left a positive pressure of 3 lb. inside the container.

As an added precaution, a "getter" layer of highly reactive magnesium-lithium alloy chips was placed in the bottom of the unit adjacent to the gas inlet pipe and covered with an asbestos paper disk.

The following conditions were observed during the initial runs:

1. In each case, specimens developed a friable "surface" layer which interrupted electrical contact before the desired temperature of 700°F was attained.
2. The "getter" material was completely consumed.
3. Thermal expansion loosened contacts slightly.
4. An acrid and heavy white vapor was given off when the

(a) Obtained from Troy Belting & Supply, Troy, N.Y.

unit was flushed during operation.

The corrosion conditions may have been due to several possible causes and it was difficult to single out the more important. It was believed, however, that certain organic binders used in the insulation and gasket materials may have been particularly harmful. On that precept, a "decontamination" run was then undertaken in which the unit was operated without specimens at 700°F for 3 days, and flushed thoroughly with helium several times each day. A considerable amount of volatile material was driven off as seen by the character of the vapors flushed out. During this operation, the brass fittings and copper in the unit developed a dark film, but the "Varglas" insulation and bonded mica were clean and bright.

Operation of the unit with specimens in place was resumed, using the same technique as before. In addition, aluminum foil was placed over each side of the gasket material to prevent direct contact with the specimen container. Each time, however, the specimens became badly corroded with a friable surface coating and electrical contact was broken before the temperature reached the desired 700°F. Operation of the equipment below approximately 500°F appeared satisfactory with no evidence of a significant degree of specimen surface attack or opening of the electrical circuits.

An x-ray diffraction analysis of the friable, white powder on the specimen surfaces was made. The material was scraped from the surface of several specimens and a sufficient amount was

gathered to prepare a specimen for an x-ray diffraction pattern. Analysis of the pattern revealed that the powdered surface coating was essentially magnesium oxide. The basis for this determination was the Hanawalt technique[1]. As a check, a pattern of high-purity magnesium oxide was prepared. The two patterns were identical for all practical purposes. These observations led to the belief that unidentified impurities in the tank helium were responsible for the condition. Provision was made to pass the tank helium through activated charcoal at the temperature of liquid nitrogen and to use an atmosphere transmission system composed entirely of copper tubing.

The completed purifying train consisted of a cylindrical copper trap, filled with sugar charcoal, C.P., and having an inlet and outlet pipe of 3/16 in. copper tubing. The inlet extended to the bottom of the trap to insure thorough contact of gas and charcoal. The size of the trap was such that it could be completely suspended in a 2 liter, narrow neck Dewar flask filled with liquid nitrogen. The gas was introduced directly into the trap from the regulator and out of the trap into the specimen container.

Several trial runs were made, using the same preparation and flushing technique as in previous runs. The "Durabla" gasket material was used and the unit was held under a positive pressure of the trap purified helium. The liquid nitrogen trap operated satisfactorily in that low temperatures could be maintained for sufficiently long times. On each occasion, however, the resistivity measurement was unsuccessful because specimen corrosion at

operative temperatures above 500°F.

Inquiry revealed that the tank helium used was a double charcoal refined welding grade with a guaranteed purity of 99.99% helium. A typical analysis indicated that the major impurity was 0.002% nitrogen and that carbon dioxide, argon and methane were present in much lower concentrations. As a result of this information it was considered unlikely that the use of a charcoal-liquid nitrogen trap for the tank helium prior to entry into the specimen container would improve the purity of the gas and experimental work on this treatment of the helium was abandoned.

Investigation of Conditions of Protective Atmosphere

An auxiliary investigation was made to obtain information on the relative effectiveness of a number of atmosphere conditions for magnesium-lithium base alloys. In this work tank helium (99.99% helium) and tank argon (99.85% argon) were used. A tube furnace was fitted with a cleaned Pyrex tube, 5/8 in. diameter; one end was equipped with a removable plug (and bleed outlet) and the opposite end was connected to the gas cylinder with clean glass tubing. Temperature was controlled by a thermocouple secured in contact with the wall of the Pyrex tube. Specimens containing a high concentration of lithium were selected from the magnesium-lithium-aluminum system and were placed in the center of the Pyrex tube length near the position of the control thermocouple. In all cases, the entire system was flushed with the gas used prior to inserting the specimen. The flushing was continued several minutes after loading the specimen. Both helium

and argon atmospheres were used under static positive pressure and steady flow conditions.

The results of this study produced the following observations:

1. Tank argon (99.85% argon) did not provide adequate protection with either static or flow conditions.
2. Tank helium (99.99% helium) was effective under steady flow but not under static pressure conditions. The low steady flow of gas may have carried away all contaminating agents as they were evolved and thereby maintained an atmosphere of higher purity than obtained under static conditions. Only an almost transparent grayish film formed on a specimen under a steady trickle-flow of helium for 48 hours. It was believed that the extent of this film would not cause difficulty in the electrical resistivity equipment operation.
3. Superior surface conditions were obtained by bright polishing before testing of extruded samples. This improvement may be related to the removal of the as-extruded surface and its attendant condition of foreign material absorbed on the surface or mechanically entrained along longitudinal die lines.

A metallographic examination of specimens exhibiting the best surface protection revealed no significant depletion of lithium in the surface zones.

Investigation of Specimen Protection with Surface Coatings

Mellor[3] reported encouraging success with the use of a proprietary mixture called "Keepbryte" for surface protection of magnesium-zirconium alloys. In communication with Mr. Mellor, it was learned that "Keepbryte" is essentially boric acid with a small amount of ferric oxide and that the mixture had been used to provide good protection for a magnesium-12 lithium-1.7 cerium alloy during solution treatment for 24 hours at 946°F.

On this information, a series of tests were undertaken to study the behavior of this material as a means of protecting magnesium-lithium base ternary alloys during extended heat treatment at elevated temperatures.

A ground mixture of 97% boric acid and 3% ferric oxide was prepared and placed in a Monel boat. 1/8 in. wire specimens of several alloys with high-lithium content were embedded in the powder and given a 700°F heat treatment. After 24 hours the specimens were removed and examined. Surface attack of the specimens was not pronounced and the stencil identification of the specimens was still readable. With these encouraging results, further trials were made with the following results:

1. A 1/32 in. layer of the boric acid-ferric oxide mixture affords adequate surface protection to specimens if it is distributed uniformly and remains unbroken.
2. The surface coating remains highly viscous at the elevated temperature but becomes brittle and glassy at room temperature.

3. Rapid heating results in severe bubbling and breaking of the protective film.

Although specimen protection with the boric acid-ferric oxide coating was demonstrated, application of the treatment to the present form of the electrical resistivity apparatus was not considered feasible. However, it is believed that this method of protection could be applied to a technique for determining electrical resistivity measurements either by direct measurement at the temperature of interest or at room temperature on quenched specimens.

Status of Development and Plans for Future Operation

The most recent observations with the unit have been made with a continuously flowing helium protective atmosphere. The result was highly encouraging in that the unit was made to operate for a period of 7 days with a gradual temperature rise to 712°F before circuit interruption. For this recent work, copper-sandwich type gaskets were used for sealing the container. Although they were not as pressure-tight as the Durabla gasket material, there was less danger of contamination from solvents and binders.

It has also been found that the high-temperature strength of the aluminum cylinder was not all that was desired and the problem of studs working out of the cylinder has arisen. This can be overcome temporarily by redrilling and tapping deeper into the wall.

Experimental work is in progress for continued operation of the unit at temperatures as near 700°F as can be obtained with

continuous flow of protective atmosphere. The most effective rate of flow of helium will be determined. It is believed that the equipment will be useful in supplementing the observations of microscopic and x-ray diffraction information for determination of phase boundaries.

# X-RAY DIFFRACTION STUDIES OF MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

## X-Ray Diffraction at Elevated Temperatures

The design, construction and initial operation of an apparatus for elevated temperature x-ray diffraction was described in the last Summary Report[1]. The intended use for this equipment in the phase boundary study was to provide a means of identifying the phases present at elevated temperatures and to supplement the microscopic examination method in locating phase boundaries.

A large number of elevated temperature diffraction patterns for a variety of magnesium-lithium-aluminum and magnesium-lithium-zinc alloys were made in the course of investigating the applicability of the method. Two major difficulties encountered in this work were (a) protection of small (nominally 0.5 mm. diameter) powder specimens from reacting with the atmosphere during elevated temperature diffraction and (b) progressive loss of lithium from the surface of specimens during diffraction. These conditions were not serious for alloys containing lower concentrations of lithium (the order of 5% or less) and at temperatures below approximately 500°F. The more serious character of these difficulties with increased lithium concentrations and temperatures produced an uncertainty in the elevated temperature diffraction results. The progress made in x-ray diffraction program is described in the following sections.

## Protective Atmosphere During Diffraction

Commercial purity tank argon (99.8% argon) was used as a protective atmosphere for experimental alloys in the x-ray diffraction work described in the last Summary Report[1]. On the basis of a considerable amount of evidence, it was concluded that this gas contained a sufficient amount of impurities to produce surface attack on specimens at 700°F. This attack was more extensive the higher the lithium concentration of the alloy.

An investigation was made to determine the effectiveness of commercial purity tank helium (welding grade double charcoal refined), 99.99% helium, in preventing specimen attack during diffraction. Specimens of beta phase alloys, L-21 (magnesium-11.85 lithium) for example, were visibly attacked to a significant degree during the diffraction cycle when maintaining a constant flow of argon through the unit. The use of helium under the same operating conditions provided excellent protection of L-21 alloy up to 700°F. The use of the helium at higher temperatures was not investigated.

In additional work using helium gas, the diffraction camera seal was sufficiently effective to permit turning off the helium after an initial flushing period. The protection achieved by this procedure was only slightly less than obtained when a positive flow of helium was maintained throughout the entire specimen heating and diffraction cycle. On the basis of these observations it was believed that helium provided a greater degree of specimen protection than argon and its use was adopted as standard practice.

Coefficients of Thermal Expansion of Experimental Alloys

An investigation was started to obtain coefficients of thermal expansion of the experimental alloys. The purpose of this work was to provide an isolation of the lattice parameter versus composition effect from the lattice parameter thermal expansion versus temperature effect.

Lattice parameter data for four different magnesium-lithium-aluminum single phase alpha solid solution alloys were plotted as a function of specimen temperature to show the thermal expansion characteristics of the close-packed hexagonal alpha structure. The values were plotted from room temperature up to 600°F for the alloys listed in Table VI.

TABLE VI

IDENTIFICATION OF ALLOYS FOR PRELIMINARY THERMAL EXPANSION STUDY

| Alloy No. | Extrusion No. | Analyses, weight percent Mg(a) | Li | Al |
|---|---|---|---|---|
| L-51 | 185 | (90.34) | 7.64 | 2.02 |
| L-55 | 190 | (93.0) | 5.1 | 1.87 |
| L-56 | 191 | (91.43) | 4.70 | 3.87 |
| L-57 | 196 | (90.30) | 3.56 | 6.14 |

(a) Magnesium analysis by difference.

The straight line curves obtained were in close agreement in slope for the different alloys, with a greater slope for the $c_o$

values than for the $a_o$ values. This indicates somewhat more rapid expansion of the hexagonal alpha phase in the axial direction than in the basal direction (Perpendicular to the hexagonal axis).

The average values of the coefficients of thermal expansion calculated from the slopes of these curves are as follows:

For $c_o$ values = $23.2 \times 10^{-6}$ per °F.

For $a_o$ values = $18.1 \times 10^{-6}$ per °F.

These may be compared with the average handbook values for the pure elements at room temperature as follows:

For magnesium = $14 \times 10^{-6}$ per °F.

For lithium = $31 \times 10^{-6}$ per °F.

For aluminum = $13.3 \times 10^{-6}$ per °F.

The average values are presented only as an indication of the approximate linear thermal expansion in the range from 75°F to 600°F for several alloys differing in composition but similar in structure and in their thermal expansion characteristics.

A tentative value of $24.1 \times 10^{-6}$ per °F was obtained for the coefficient of expansion of the body-centered cubic beta phase for the binary alloy L-21, containing 11.85% lithium balance magnesium, in the range from 75°F to 500°F.

Additional Experience with Elevated Temperature Diffraction

More lattice parameters were obtained from the x-ray diffraction data and it was planned to analyze the values more completely to determine the specific effects of composition and phase structure on the coefficients of thermal expansion for the various alloys. As the study progressed it was found that there was a

general lack of reproducibility of lattice parameters determined on the same alloy several months apart. The lattice parameters of two single phase alpha structures, for example, illustrate this observations. These alloys were:

L-55 magnesium-5.1 lithium-1.87 aluminum

L-56 magnesium-4.7 lithium-3.87 aluminum.

All patterns for alloy L-55 were prepared in a commercial argon atmosphere from room temperature to 600°F. The room temperature and 300°F patterns of L-56 were prepared in a commercial argon atmosphere, whereas patterns at 500°F and 700°F were made in a commercial helium atmosphere. It was believed that the discrepancy in results may have been due to a loss of lithium from the metal during storage, specimen preparation or diffraction periods. In addition, the results of a combination of microscopic and diffraction evidence for several alloys indicated the possibility for a complete misinterpretation of the representative structure by x-ray diffraction where a loss of lithium from the surface of specimens was not prevented.

Due to the evidence that loss of lithium occurred from the x-ray specimen at elevated temperatures, it was believed that a thorough investigation should be made of the degree of the loss and methods of its prevention before results from the x-ray diffraction technique could be trusted to locate phase boundaries. Therefore, in order to devote more time to the microscopic examination method for location phase boundaries, experimental work on the x-ray diffraction problems related to establishing phase boun-

daries was discontinued temporarily.

Identification of Intermediate Phases by X-Ray Diffraction

An investigation is in progress to establish the identity of intermediate phases encountered in the magnesium corner of magnesium-lithium-aluminum and magnesium-lithium-zinc systems. A series of alloys was prepared, the compositions of which were intended to be specific intermediate phases whose presence is anticipated or suspected in the phase fields of interest. A list of the phases is given in Table VII.

TABLE VII

INTENDED COMPOSITIONS OF INTERMEDIATE PHASES PREPARED TO SERVE AS IDENTIFICATION STANDARDS

| Phase | Atomic Percent | | | | Weight Percent | | | |
|---|---|---|---|---|---|---|---|---|
| | Mg | Li | Al | Zn | Mg | Li | Al | Zn |
| AlLi | | 50 | 50 | | | 21 | 79 | |
| $AlLi_2$ | | 67 | 33 | | | 34 | 66 | |
| $Mg_{17}Al_{12}$ | 57 | | 43 | | 55 | | 45 | |
| $MgLi_2Al$ | 25 | 50 | 25 | | 37.3 | 21.3 | 41.4 | |
| $MgLiAl_2$ | 25 | 25 | 50 | | 29.9 | 10.3 | 59.8 | |
| MgZn | 50 | | | 50 | 27.1 | | | 73.9 |
| MgLiZn | 33 | 33 | | 33 | 25.3 | 7.2 | | 67.5 |
| $MgLi_2Zn$ | 25 | 50 | | 25 | 23.4 | 13.4 | | 63.2 |

## Status of Diffraction Work and Plans for Future

The preparation of x-ray diffraction patterns and determination of lattice type and parameter for intermediate phases in the magnesium-lithium-aluminum and magnesium-lithium-zinc systems is in progress. At present these patterns are being determined at room temperature. These patterns and their data characteristic of specific phases will be used later to establish the identity of intermediate phases in the ternary alloys in the magnesium corner of the two systems.

Most of the intermediate phase alloys prepared contain a large concentration of lithium and are distinctly reactive at room temperature in air. Powdering of the brittle alloys in air and under a bath of kerosene has been tried. These methods have not been entirely successful and it is believed to be necessary to carry out the entire operation of powdering, screening, mixing powders into a paste with collodion and extrusion to a cylindrical wire specimen in an inert atmosphere. A large dry box has been prepared for this operation and the operation will be tried using helium as a protective atmosphere.

In addition, it is planned to continue the study of methods for the prevention of specimen reaction during elevated temperature diffraction so that this method of locating phase boundaries can be used.

# DEVELOPMENT OF LOW ALLOY CONTENT HIGH DUCTILITY MAGNESIUM BASE ALLOYS

## Experimental Procedure for Alloy Preparation and Evaluation

### Objective of the Research

The principal objective of this development was to produce alloys with an optimum combination of strength and formability in sheet form, using total alloying additions of the order of one percent and some combination of mechanical and thermal treatment.

### Results of Initial Work on the Problem

Initial work on this problem investigated the mechanical properties of alloys in five separate systems:

magnesium-zinc-cerium

magnesium-aluminum-cerium

magnesium-cerium

magnesium-zirconium

magnesium-zinc-zirconium.

The results of the experimental work in these systems, reported in the last Summary Report[1], gave the following indications:

1. A relatively high combination of strength, elongation and toughness for several alloys in the magnesium-zinc-cerium system justified a more thorough study of this system.

2. Mechanical properties of alloys in the systems magnesium-aluminum-cerium and magnesium-cerium were not of sufficient interest to merit additional study.

3. A relatively low order of retention of zirconium in magnesium-zirconium and magnesium-zinc-zirconium alloys was encountered. It was considered, therefore, that the mechanical properties obtained for alloys in these two systems were not representative of the capabilities of the systems. It was recommended that the experimental work in the two systems be repeated with better methods for the introduction and retention of zirconium.

Experimental Alloy Selection, Preparation and Evaluation

A major share of the experimental work described in the present report on the general problem of developing low alloy content, high ductility alloys was devoted to a study of alloys in the magnesium-zinc-cerium system. Two additional methods of introducing zirconium into magnesium were studied and were applied successfully.

The experimental procedures used in alloy preparation and evaluation are summarized in Appendix VI. A summary of intended compositions, spectrographic analyses, mechanical and thermal treatments for all experimental alloys prepared is given in Appendix VII.

A standardized procedure for alloy fabrication was developed. This procedure was intended to survey the capabilities of the alloys and predict compositions warranting a more complete

study. Two methods of sheet rolling were used as specified in the following designations:

1. hot rolling - rolling alloys at a temperature range believed to be above the recrystallization temperature range for the conditions involved.
2. warm rolling - rolling alloys at an elevated temperature believed to be below the recrystallization temperature range for the conditions involved.

After rolling, recrystallization curves were determined for both types of sheet and the mechanical properties were determined in three conditions of thermal treatment. These conditions are identified, together with the specimen nomenclature used throughout the work in the following list:

H - hot rolled sheet

W - warm rolled sheet

s - stress relieved after rolling, at a temperature near the lower limit of the recrystallization temperature range for one hour.

a(L) - low temperature annealing treatment after rolling, at a temperature near the middle of the recrystallization temperature range for one hour.

a(H) - high temperature annealing treatment after rolling, at a temperature near the upper limit

of the recrystallization temperature range for one hour.

A standardized schedule for sheet fabrication and testing is given in Fig. 14. The experimental details of sheet fabrication and testing are given in Appendices VI and VII.

## Magnesium-Zinc-Cerium Alloys

### Review of Previous Investigation

The results of an initial study of the mechanical properties of alloys in this system with zinc and cerium ranges between 0 and 1.0% were given in the last Summary Report[1]. This work indicated that the best composition in the range studied was magnesium-0.8 zinc-0.2 cerium. The average mechanical properties for this alloy are given in Tables VIII and IX. These data indicated that an attractive combination of high elongation and relatively high strength was produced by the procedure of warm rolling and stress relieving. A metallographic examination of these demonstrated that the greatest improvement in mechanical properties was associated with an extremely fine, uniform, equiaxed recrystallized grain structure - a structure produced by warm rolling and stress relieving heat treatment.

These results were considered to be sufficient evidence to warrant a more thorough study of the magnesium-zinc-cerium system. The scope of continued investigation is given in the next section.

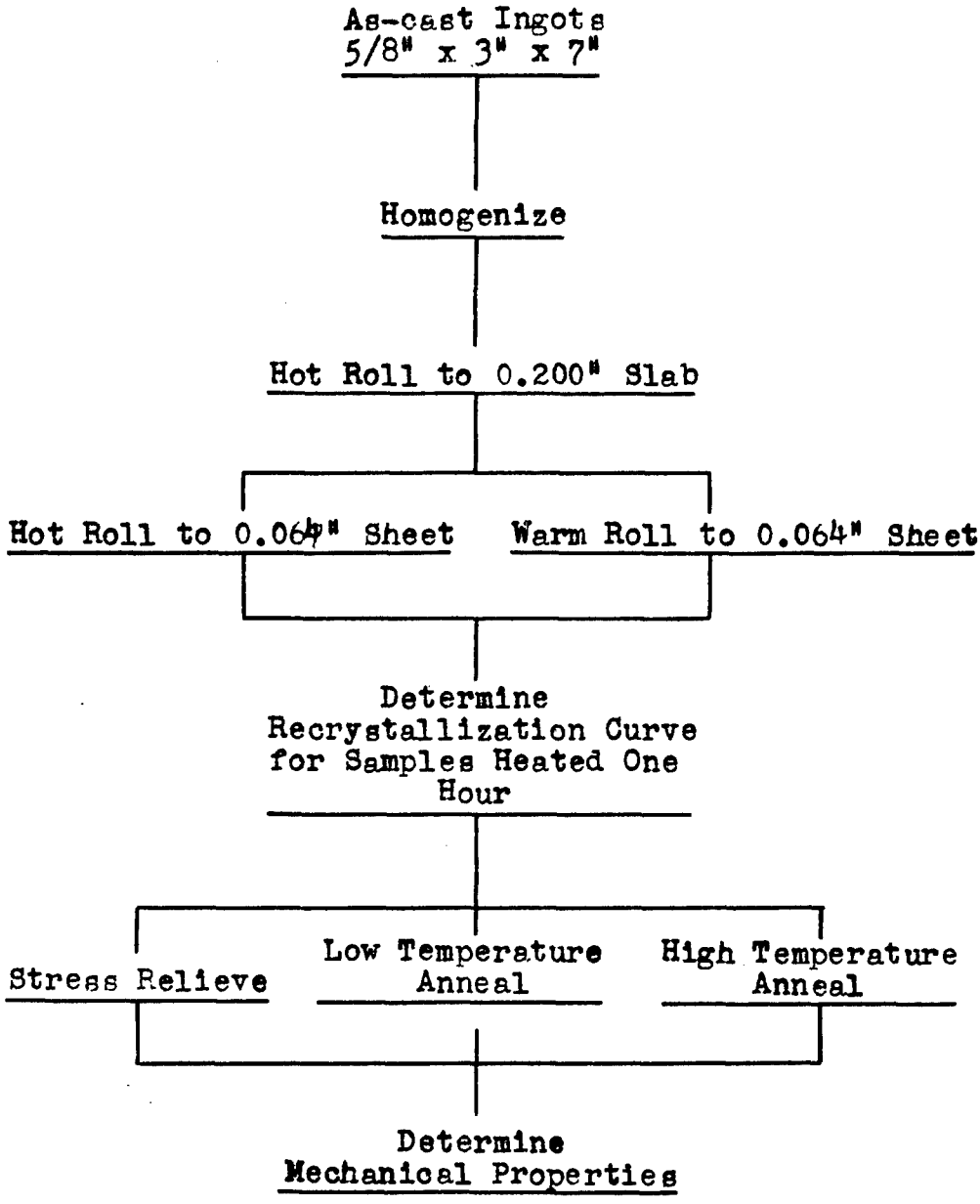



Figure 14

Standardized Schedule for Survey of Mechanical Properties of Experimental Low Alloy Content Magnesium-Base Alloys.

TABLE VIII

AVERAGE MECHANICAL PROPERTIES OF MAGNESIUM-

0.8 ZINC-0.2 CERIUM ALLOY

| Alloy | Nominal Rolling Temp., °F Hot | Warm | Heat Treatment Time | Temp. | Final Cold Reduction | Kips per sq.in.(a) CYS | TYS | UTS | Elong. % in 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| R-614Hs | 700 | - | 1 | 600°F | - | 20.0 | 23.6 | 33.0 | 10.0 |
| 614Ha(H) | 700 | - | 1 | 750 | - | 12.1 | 16.2 | 29.9 | 14.5 |
| 612Hc | 700 | - | - | - | 15% | 22.3 | 31.0 | 36.6 | 4.0 |
| 613Ws 669Ws | 700 | 400 | 1 | 600 | - | 14.6 | 24.3 | 34.3 | 21.0 |
| 613Wa(H) 669Wa(H) | 700 | 400 | 1 | 750 | - | 13.2 | 22.9 | 33.9 | 20.0 |
| 613Wc | 700 | 400 | - | - | 15 | 34.3 | 38.4 | 43.7 | 1.5 |

(a) Average of at least three samples for each condition.

TABLE IX

AVERAGE NOTCH SENSITIVITY PROPERTIES OF MAGNESIUM-

0.8 ZINC-0.2 CERIUM ALLOY

| Alloy | Ultimate Torque in.-lb. | Ultimate Bend Angle Degrees | Static Rupture Energy in.-lb. | Strength Factor | Ductility Factor | Toughness Factor |
|---|---|---|---|---|---|---|
| R-614Hs | 19.6 | 80.0 | 22.60 | 0.593 | 6.40 | 0.0547 |
| 614Ha(H) | 16.4 | 95.0 | 22.00 | 0.548 | 6.55 | 0.0507 |
| 613Ws 669Ws | 19.4 | 67.6 | 18.94 | 0.565 | 3.08 | 0.0252 |
| 613Wa(H) 669Wa(H) | 18.7 | 67.8 | 17.99 | 0.552 | 3.46 | 0.0237 |

## Scope of Continued Investigation of the System

Additional work on magnesium-zinc-cerium alloys was divided into two parts. These were:

1. A study of the effect of small variations in zinc and cerium using the nominal composition magnesium-0.8 zinc-0.2 cerium as a base.
2. A study of the effect of large variations in zinc and cerium with zinc additions ranging up to 3.0% and cerium additions ranging up to 1.5%.

## Investigation of Small Variations in Zinc and Cerium Additions

In preparation for the general evaluation of the effects of small variations in zinc and cerium additions, a preliminary study was made to select a temperature for hot rolling and to determine whether or not the beneficial effects of the warm rolling procedure could be duplicated by cold rolling the hot rolled sheet prior to heat treatment. The conditions were selected to:

1. Compare the effect of hot rolling at 700°F and at 750°F on the mechanical properties, using a standardized schedule for sheet preparation.
2. Compare the following two methods of finishing sheet on the annealed and stress relieved mechanical properties:
   a. Hot roll to 0.075", heat one hour at the rolling temperature and cold roll approximately 15% to 0.064" sheet.
   b. Hot roll to 0.200" slab and then warm roll at

400°F to 0.064" sheet.

For this preliminary study, the composition magnesium-0.8 zinc-0.2 cerium was used in order to compare the results with the favorable mechanical properties developed by this alloy in the previous study.

The mechanical properties from the preliminary study are listed in Table X. For identical treatments these data indicated that 700°F should be used for rolling and this temperature was selected for hot rolling the remaining compositions of the group.

The mechanical properties of R-613, 614 and 669, magnesium-0.8 zinc-0.2 cerium from the original work on this system, are included in Table X for comparison with the new data. Strict adherence to the belief that optimum mechanical properties would result from a stress relieving treatment just short of producing recrystallization was the basis for selecting 400°F from recrystallization curves in this preliminary study. This treatment resulted in consistently lower elongations than obtained in the original work. To check these results, the third specimen of R-739Ws was given an additional 1 hour treatment at 600°F. It was encouraging to find that the resulting mechanical properties duplicated the values of R-613Ws and R-669Ws. On the basis of this observation it is believed that the optimum condition is a completely recrystallized, extremely fine grained internal structure. This principle was applied to all additional work on the system.

TABLE X

MECHANICAL PROPERTIES OF MAGNESIUM-0.8 ZINC-0.2 CERIUM ALLOYS TO ESTABLISH OPTIMUM CONDITIONS FOR SHEET PREPARATION

| Alloy | Nominal Rolling Temp. °F Hot | Warm | Cold Reduction Prior to Heat Treatment | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % in 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| R-739Ha | 700 | - | - | 1 hr. | 750°F | 12.1 | 19.5 | 30.2 | 10.0 (a) |
| | | | | | | 12.7 | 18.0 | 38.9 | 9.0 (a) |
| | | | | | | 12.7 | 17.9 | 29.9 | 10.0 |
| | | | | (average) | | 12.5 | 18.5 | 29.7 | 9.7 |
| R-739Hs | 700 | - | - | 1 | 400 | 27.4 | 31.8 | 36.7 | 12.0 |
| | | | | | | 26.3 | 31.1 | 37.1 | 12.0 |
| | | | | | | 25.8 | 31.1 | 37.3 | 12.0 |
| | | | | (average) | | 26.5 | 31.3 | 37.0 | 12.0 |
| R-739Hca | 700 | - | 13% | 1 | 750 | 11.3 | 14.2 | 28.5 | 9.0 (a) |
| | | | | | | 10.7 | 15.2 | 27.4 | 7.0 (a) |
| | | | | | | 10.8 | 14.9 | 27.1 | 7.0 (a) |
| | | | | (average) | | 10.9 | 14.8 | 27.7 | 7.7 |
| R-739Hcs | 700 | - | 13 | 1 | 400 | 20.4 | 28.6 | 35.4 | 10.0 (a) |
| | | | | | | 20.4 | 28.3 | 35.4 | 10.0 (a) |
| | | | | | | 20.6 | 29.5 | 35.9 | 11.0 |
| | | | | (average) | | 20.5 | 28.8 | 35.6 | 10.3 |
| R-739Wa | 700 | 400 | -- | 1 | 750 | 12.7 | 19.7 | 31.7 | 10.0 |
| | | | | | | 13.7 | 19.3 | 31.4 | 9.0 |
| | | | | | | 12.9 | 19.6 | 30.8 | 9.0 |
| | | | | (average) | | 13.1 | 19.5 | 31.3 | 9.3 |
| R-739Ws | 700 | 400 | - | 1 | 400 | 25.8 | 30.5 | 39.9 | 5.0 |
| | | | | | | 25.5 | 30.1 | 38.0 | 3.0 (a) |
| | | | PLUS | 1 | 600 | 18.0 | 26.5 | 35.7 | 21.0 (b) |
| R-614Ha | 700 | - | - | 1 | 750 | 12.1 | 16.2 | 29.9 | 14.5 (c) |
| R-614Hs | 700 | - | - | 1 | 600 | 20.0 | 23.6 | 33.0 | 10.0 (c) |
| R-613Wa R-669Wa | 700 | 400 | - | 1 | 750 | 13.2 | 22.9 | 33.9 | 20.0 (c) |

(a) Specimen fractured through a tensometer notch.
(b) Specimen given an additional treatment of 1 hr. at 600°F.
(c) Average mechanical properties.

TABLE X, Cont'd

| Alloy | Nominal Rolling Temp. °F Hot | Warm | Cold Reduction Prior to Heat Treatment | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % in 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| R-613Ws R-669Ws | 700 | 400 | - | 1 | 600°F | 14.6 | 24.3 | 34.3 | 21.0(c) |
| R-738Ha | 750 | - | - | 1 | 750 | 9.2 | 14.9 | 29.4 | 8.0(a) |
| | | | | | | 9.5 | 14.9 | 29.0 | 8.0 |
| | | | | | | 9.5 | 15.4 | 28.8 | 8.0 |
| | | | | (average) | | 9.4 | 15.1 | 29.1 | 8.0 |
| R-738Hs | 750 | - | - | 1 | 400 | 17.3 | 24.6 | 31.5 | 10.0 |
| | | | | | | 18.6 | 24.6 | 31.7 | 8.5 |
| | | | | | | 17.1 | 24.8 | 32.5 | 7.0 |
| | | | | (average) | | 17.7 | 24.7 | 31.9 | 8.5 |
| R-738Hca | 750 | - | 13% | 1 | 750 | 8.1 | 13.4 | 26.2 | 5.7 |
| | | | | | | 8.7 | 13.3 | 24.8 | 5.0(a) |
| | | | | | | 8.7 | 13.4 | 27.0 | 6.0 |
| | | | | (average) | | 8.5 | 13.4 | 26.2 | 5.7 |
| R-738Hcs | 750 | - | 13 | 1 | 400 | 15.8 | 24.5 | 31.4 | 5.0(a) |
| | | | | | | 15.8 | 22.7 | 31.6 | 4.0 |
| | | | | | | 16.0 | 23.0 | 31.6 | 4.0 |
| | | | | (average) | | 15.9 | 23.4 | 31.5 | 4.3 |
| R-738Wa | 750 | 400 | - | 1 | 750 | 9.8 | 17.3 | 29.5 | 6.0 |
| | | | | | | 10.7 | 16.1 | 28.3 | 5.0(a) |
| | | | | | | - | 16.8 | 29.5 | 6.0 |
| | | | | (average) | | 10.3 | 16.7 | 29.1 | 5.7 |
| R-738Ws | 750 | 400 | - | 1 | 400 | 25.9 | 34.3 | 39.8 | 9.0(a) |
| | | | | | | 25.8 | 32.0 | 40.0 | 11.0 |
| | | | | | | - | 32.4 | 39.2 | 3.0 |
| | | | | (average) | | 25.85 | 32.9 | 39.7 | 7.7 |

(a) Specimen fractured through tensometer notch.

The data in Table X show that an excellent combination of mechanical properties can be obtained by warm rolling as the procedure prior to the final thermal treatment. Although the practice of cold rolling prior to thermal treatment did not produce favorable results, it was considered that the extent of the study was too limited to conclude that the procedure was unsuitable as a substitute for the warm rolling practice. At this stage of the study it was not considered advisable to investigate the method more completely and the warm rolling practice was continued as a standard condition.

A series of 15 compositions in the nominal range magnesium-0.8 zinc-0.2 cerium was melted and cast. The intended compositions and spectrographic analyses of these alloys are listed in Table XI. Since the analyses were different from the intended compositions the results of the study are presented and interpreted in terms of these analyses. It was found that the most informative analysis of the data was obtained by considering the mechanical properties in relation to the ratio of cerium to zinc. The average mechanical properties of warm and hot rolled sheet are listed according to the cerium/zinc ratio in Table XII. The condition of high temperature annealing was removed as a variable in the evaluation because of the generally lower level of a combination of strength and elongation properties.

The data in Table XII are presented in graphical form in Figs. 15, 16, 17 and 18. In Figs. 15 and 16 are plotted the elongation values and strength properties, respectively, for warm

TABLE XI

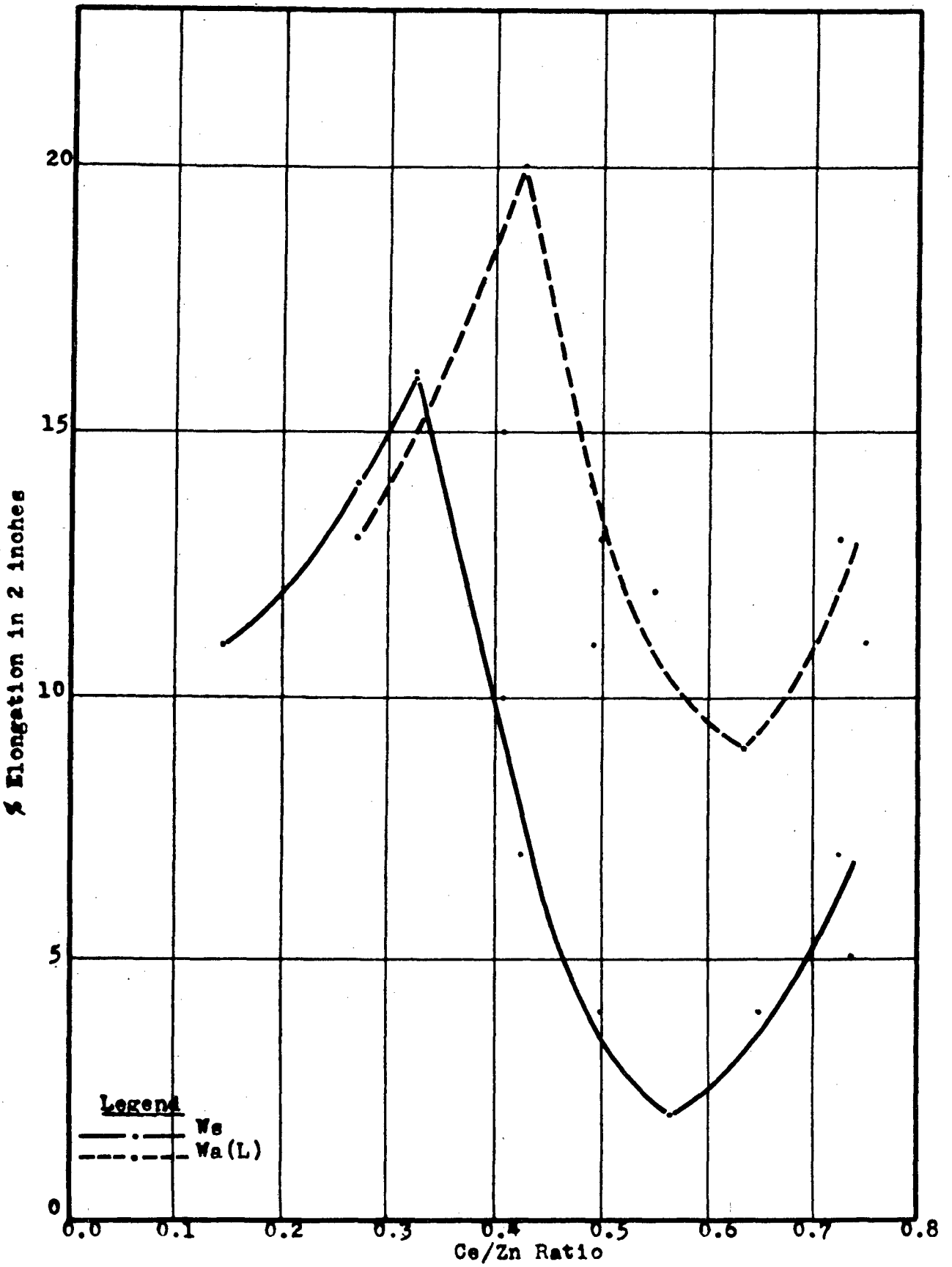
INTENDED COMPOSITIONS AND SPECTROGRAPHIC ANALYSES OF MAGNESIUM-ZINC-CERIUM ALLOYS FOR SMALL VARIATIONS IN COMPOSITION SURVEY

| Alloy | Intended Composition | | Spectrographic Analyses | |
|---|---|---|---|---|
| | Zinc | Cerium | Zinc | Cerium |
| R-750 | 0.6 | - | 0.61 | - |
| 728 | 0.7 | - | 0.55 | - |
| 729 | 0.8 | - | 0.64 | - |
| 730 | 0.6 | 0.1 | 0.62 | 0.09 |
| 760 | 0.7 | 0.1 | 0.53 | 0.18 |
| 757 | 0.8 | 0.1 | 0.78 | 0.21 |
| 761 | 0.6 | 0.2 | 0.46 | 0.23 |
| 747 | 0.7 | 0.2 | 0.54 | 0.23 |
| 756 | 0.8 | 0.2 | 0.68 | 0.22 |
| 754 | 0.6 | 0.3 | 0.51 | 0.37 |
| 755 | 0.7 | 0.3 | 0.57 | 0.28 |
| 742 | 0.8 | 0.3 | 0.76 | 0.31 |
| 743 | 0.6 | 0.5 | 0.62 | 0.35 |
| 744 | 0.7 | 0.5 | 0.68 | 0.50 |
| 745 | 0.8 | 0.5 | 0.74 | 0.47 |

TABLE XII

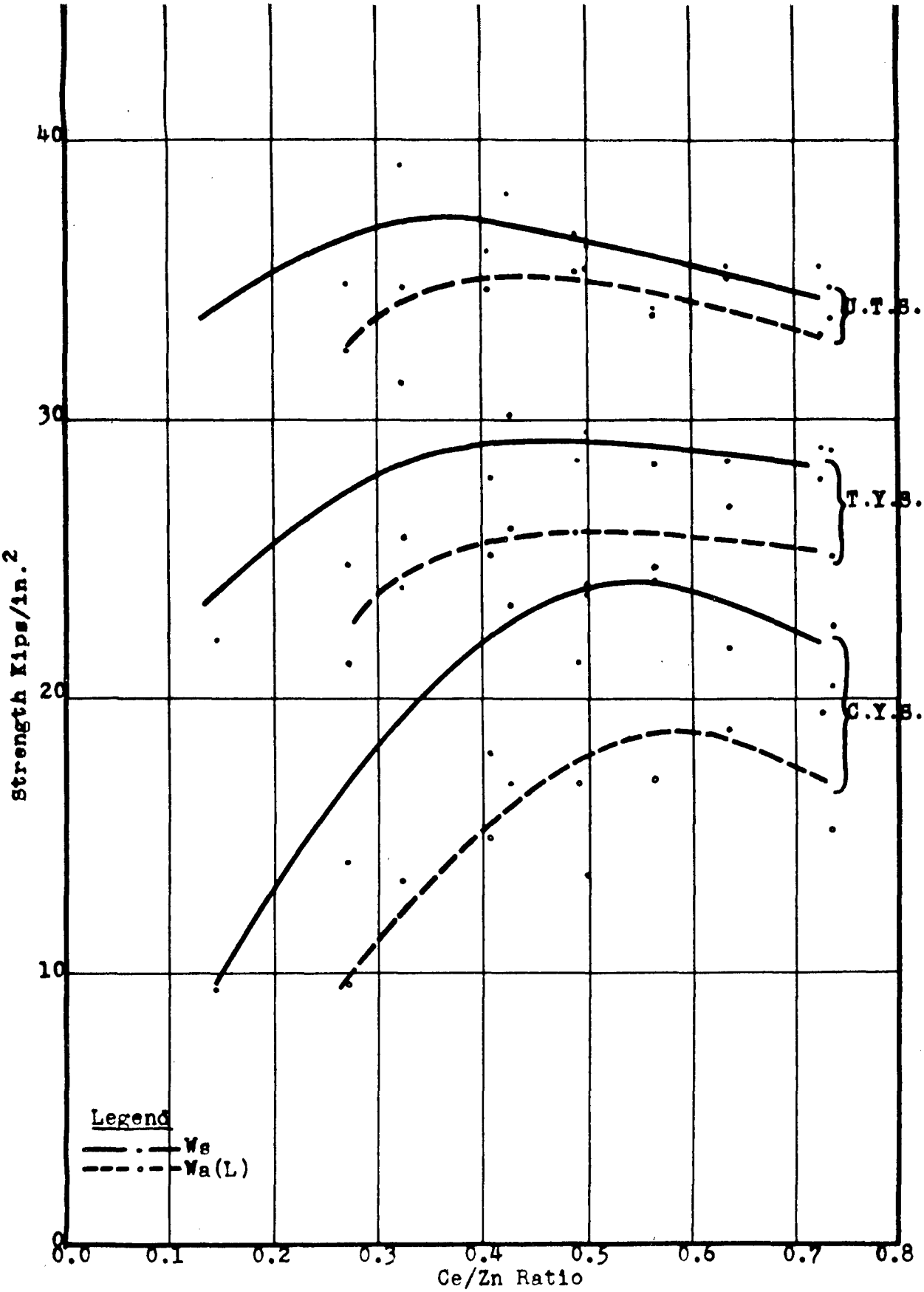
AVERAGE MECHANICAL PROPERTIES OF WARM AND HOT ROLLED MAGNESIUM-ZINC-CERIUM ALLOYS FOR SMALL VARIATIONS IN COMPOSITION STUDY LISTED ACCORDING TO RATIO OF CERIUM/ZINC SPECTROGRAPHIC ANALYSES

| | | Warm Rolled and Stress Relieved (Ws) | | | | Warm Rolled and Low Temperature Annealed (Ws(L)) | | | | Hot Rolled and Stress Relieved (Hs) | | | | Hot Rolled and Low Temperature Annealed (Ha(L)) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Kips/sq.in. | | | Elong. | Kips/sq. in. | | | Elong. | Kips/sq.in. | | | Elong. | Kips/sq.in. | | | E |
| Alloy | Ce/Zn Ratio | CYS | TYS | UTS | % 2 in. | CYS | TYS | UTS | % 2 in. | CYS | TYS | UTS | % 2 in. | CYS | TYS | UTS | % |
| R-730 | 0.145 | 9.4 | 22.1 | 32.3 | 11 | - | - | - | - | 7.9 | 20.1 | 32.3 | 7 | - | - | - | |
| 757 | 0.270 | 14.0 | 24.8 | 34.9 | 14 | 9.6 | 21.3 | 32.5 | 13 | 10.9 | 23.0 | 32.4 | 11 | 7.6 | 21.1 | 32.3 | |
| 756 | 0.324 | 24.1 | 31.3 | 39.1 | 16 | 13.3 | 25.8 | 34.8 | 16 | 8.5 | 14.9 | 28.6 | 10 | 7.7 | 13.1 | 27.4 | |
| 742 | 0.407 | 18.0 | 27.9 | 36.1 | 10 | 14.9 | 25.2 | 34.7 | 15 | 18.3 | 28.3 | 37.1 | 21 | 15.6 | 21.0 | 33.5 | |
| 747 | 0.426 | 23.4 | 30.1 | 38.1 | 7 | 16.9 | 26.1 | 35.2 | 20 | 13.8 | 21.4 | 30.4 | 10 | 9.1 | 13.6 | 28.5 | |
| 755 | 0.492 | 21.3 | 28.5 | 36.7 | 11 | 16.9 | 26.0 | 35.4 | 14 | 21.4 | 30.8 | 37.9 | 11 | 16.3 | 23.9 | 34.7 | |
| 761 | 0.500 | 23.8 | 29.5 | 36.3 | 4 | 13.5 | 24.1 | 35.5 | 13 | 11.6 | 21.4 | 30.2 | 12 | 9.2 | 13.4 | 27.5 | |
| 743 | 0.564 | 24.2 | 28.4 | 34.0 | 2 | 17.0 | 24.8 | 33.8 | 12 | 21.9 | 30.6 | 36.8 | 4 | 15.1 | 23.1 | 34.2 | |
| 745 | 0.635 | 21.9 | 28.5 | 35.6 | 4 | 18.9 | 26.9 | 35.2 | 9 | 23.6 | 32.1 | 38.4 | 7 | 19.1 | 30.5 | 38.4 | |
| 754 | 0.725 | 25.2 | 29.0 | 35.6 | 7 | 19.4 | 27.9 | 33.1 | 13 | 19.6 | 30.3 | 36.0 | 5 | 15.6 | 23.6 | 33.8 | |
| 744 | 0.735 | 20.4 | 28.9 | 34.8 | 6 | 15.2 | 22.6 | 33.7 | 11 | 20.8 | 32.3 | 38.0 | 6 | 16.0 | 23.5 | 34.1 | |



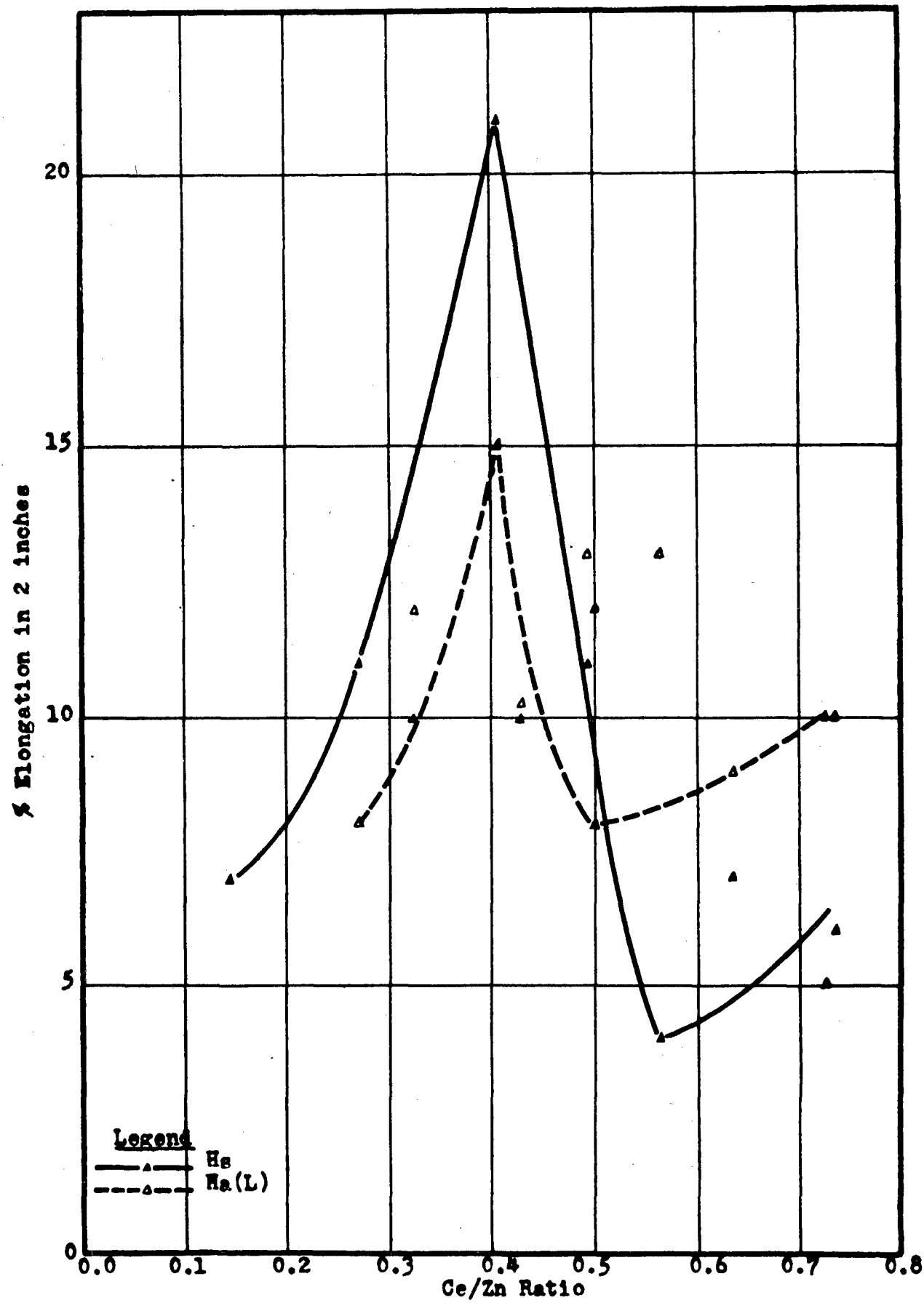

Elongation versus Ce/Zn Ratio for Warm Rolling

FIGURE 15



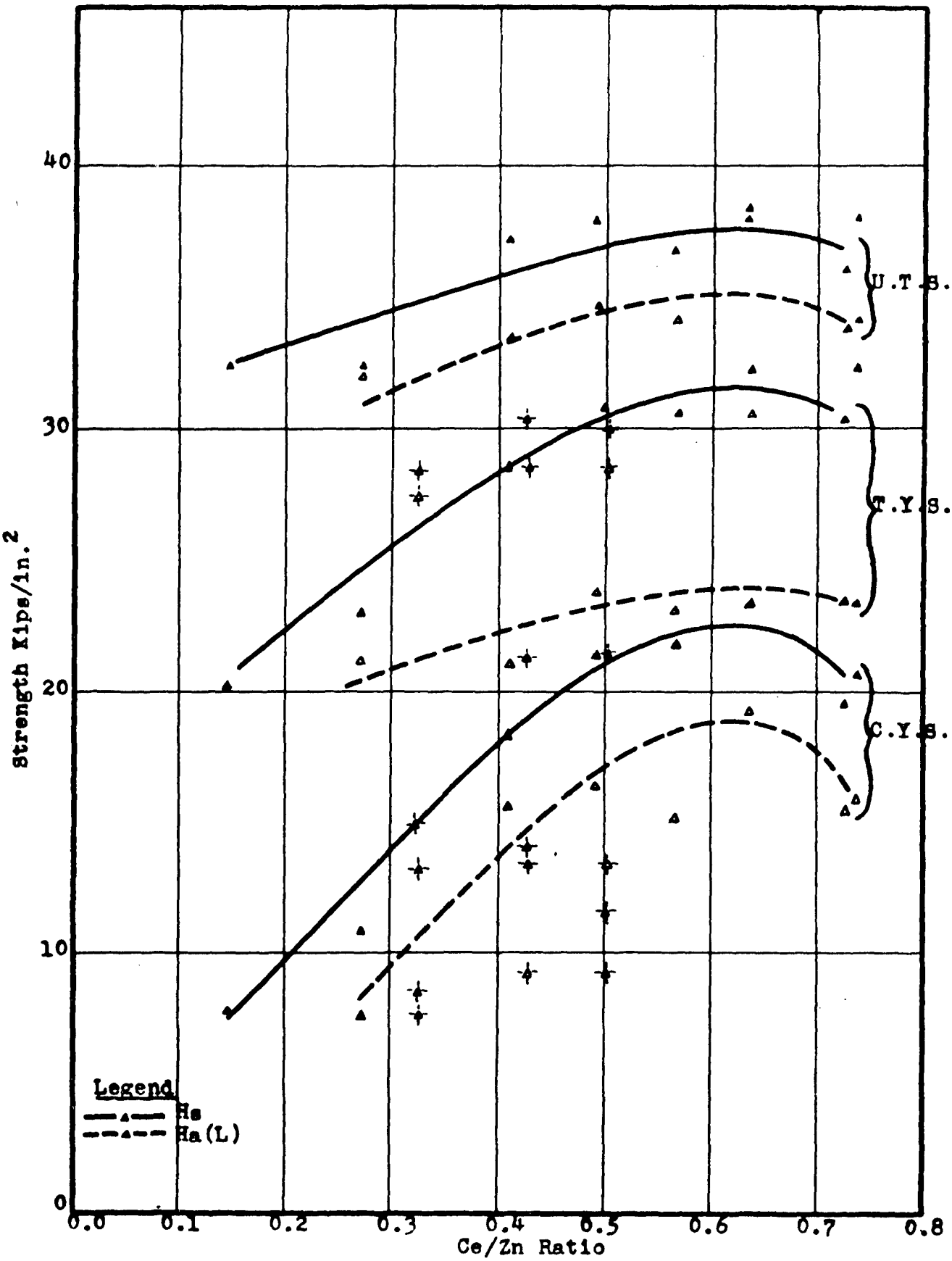

Strength Properties versus Ce/Zn Ratio for Warm Rolling

FIGURE 16




Elongation versus Ce/Zn Ratio for Hot Rolling

FIGURE 17




Strength Properties versus Ce/Zn Ratio for Hot Rolling

FIGURE 18

rolled sheet as a function of the cerium/zinc ratio. Figs. 17 and 18 present the same information for hot rolled sheet.

For warm rolled sheet, Fig. 15 shows maximum values of elongation at distinct cerium/zinc ratios. The range between the maxima for stress relieved and low temperature annealed conditions predicts a high level of elongation for a range of compositions for warm rolled sheet. These data indicate that low temperature annealing consistently produced a higher level of elongation values than stress relieving in warm rolled sheet. As shown in Fig. 16 for warm rolled sheet the ultimate and yield strengths in tension as well as the elongation reached maximum values at approximately the same cerium/zinc ratio. Compressive yield strength values reached a maximum at a slightly higher cerium/zinc ratio.

The high level of elongation produced by warm rolling and low temperature annealing was reproduced by a stress relieving treatment of hot rolled sheet at approximately the same cerium/zinc ratios as shown in Fig. 17. Low temperature annealing seriously lowered the elongation values for the hot rolled sheet. Maxima for the two thermal treatments of the hot rolled sheet were coincident at approximately 0.4 cerium/zinc ratio. Hot rolled sheet strength values (Fig. 17) reached a maximum level at considerably higher range of cerium/zinc than indicated for maximum elongation values. There was a distinct scatter of strength values at and around the optimum cerium/zinc ratio for maximum elongations, presumably due to variations in the temperatures

used for thermal treatment and to a lower degree of strain hardening during working than was developed by warm rolling.

A metallographic examination was made of representative alloys to observe the microstructural conditions associated with variations in cerium/zinc ratios, mechanical working and thermal treatments. This investigation was not comprehensive but the observations were considered significant to an interpretation of mechanical properties.

A secondary phase was observed to some extent in all alloys examined and this phase increased in amount with an increasing cerium/zinc ratio. The identity of the phase was not established. The phase was generally globular in shape and randomly distributed. Occasionally it was present as stringers of tiny particles lined up in the direction of rolling. Phospho-picral and acetic-picral etching reagents did not attack the phase but usually heavily outlined the particles from the matrix. There was some evidence to indicate, especially for the higher cerium/zinc ratios, that at least two secondary phases may have been present. The influence of an increasing cerium/zinc ratio from 0.27 to .64 on the extent of the white secondary phase is shown in Figs. 19, 20 and 21.

The distribution of the undissolved phase appeared to be the same for comparable conditions in warm and hot rolled sheet but was influenced considerably by the temperature of heat treatment. For a given composition in the stress relieved condition there was present a finely divided white phase randomly

Figure 19

Neg: 227 Mag: 500X
Spec: M663-2 Etch: Phospho-Picral, 15 sec

Longitudinal section from 757Hs (magnesium-0.78 zinc-0.21 cerium, cerium/zinc.27) hot rolled at 700°F and stress relieved 1 hour at 400°F. A few tiny particles of a white phase are visible.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 10.2 | 23.5 | 32.5 | 11 |

Figure 20

Neg: 228 Mag: 500X
Spec: M655-2 Etch: Phospho-Picral, 15 sec

Longitudinal section from 742Hs (magnesium-0.76 zinc-0.31 cerium, cerium/zinc-0.41) hot rolled at 700°F and stress relieved 1 hour at 600°F. A randomly distributed, finely divided phase and more massive particles were present.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 18.3 | 27.7 | 37.0 | 22 |

Figure 21

Neg: 226 Mag: 500X
Spec: M667-4 Etch: Phospho-Picral, 15 sec

Longitudinal section of 745Hs (magnesium-0.74 zinc-0.47 cerium, cerium/zinc-0.64) hot rolled at 700°F and stress relieved 1 hour at 550°F. The amount of undissolved phase, coarse and fine particles, was considerably greater than in Fig. 7.

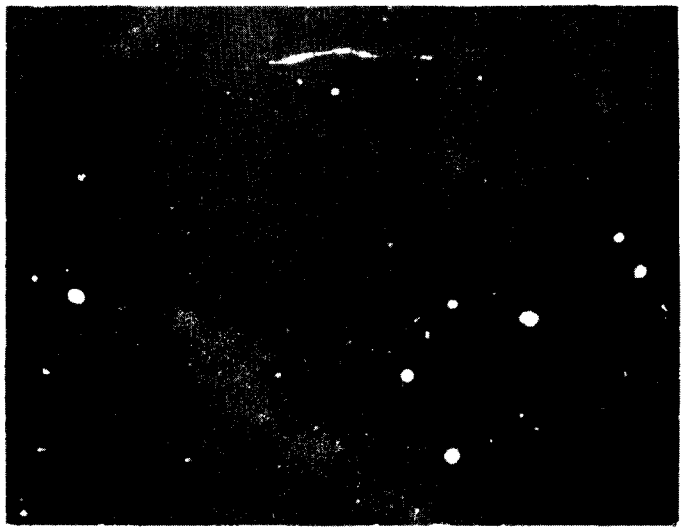
| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 23.5 | 31.8 | 38.2 | 7 |

scattered throughout the matrix in addition to a random distribution of more massive particles and stringers of undissolved phase. Heat treatments above 650°F resulted in a solution of the fine particles or agglomeration into larger particles. Although the solid solubility of undissolved phase appeared to increase with increasing temperature, even 0.31% cerium (cerium/zinc ratio .41) exceeded the solubility for a 1 hour treatment at 750°F. These conditions are shown in Figs. 22, 23 and 24 for R-742, magnesium-0.76 zinc-0.31 cerium (0.41 cerium/zinc ratio), and they were more pronounced for the 0.64 cerium/zinc ratio.

All alloys and treatments produced relatively fine grained structures ranging from considerably less than 0.00022" to approximately 0.00042" average size. The finest grain structures were associated with stress-relieving treatments and higher cerium analyses. In the stress relieved conditions for cerium/zinc ratios of 0.41 and above, there was some question as to whether the degree of recrystallization was complete in warm or hot rolled sheet. Low and high temperature annealing resulted in progressive grain coarsening compared to stress relieving. Examples of these conditions are shown in Figs. 25, 26 and 27 for hot rolled sheet and Figs. 28, 29 and 30 for warm rolled sheet.

There was some evidence to indicate that a white phase was present along grain boundaries as well as randomly distributed throughout the matrix for cerium/zinc ratios of 0.41 and above. This condition was observed in both warm and hot rolled sheet, being more pronounced with higher heat treatment tempera-

Figure 22

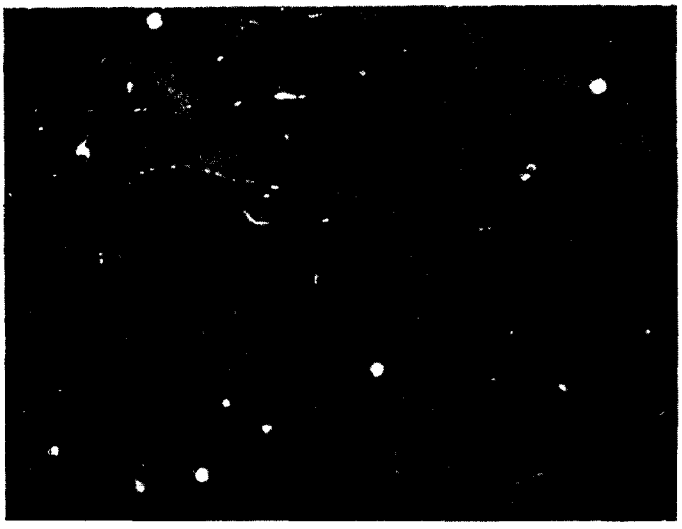


Neg: 228 Mag: 500X
Spec: M655-2 Etch: Phospho-Picral, 15 sec

Longitudinal section of 742Hs (magnesium-0.76 zinc-0.31 cerium, cerium/zinc-.41) hot rolled at 700°F and stress relieved 1 hour at 400°F. Note the general distribution of the finely divided secondary phase.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 18.3 | 27.7 | 37.0 | 22 |

Figure 23



Neg: 229 Mag: 500X
Spec: M657-2 Etch: Phospho-Picral, 15 sec

Longitudinal section of 742Ha(L), low temperature annealed 1 hour at 650°F. A large part of the finely divided phase apparently dissolved in matrix.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 15.6 | 21.0 | 33.5 | 15 |

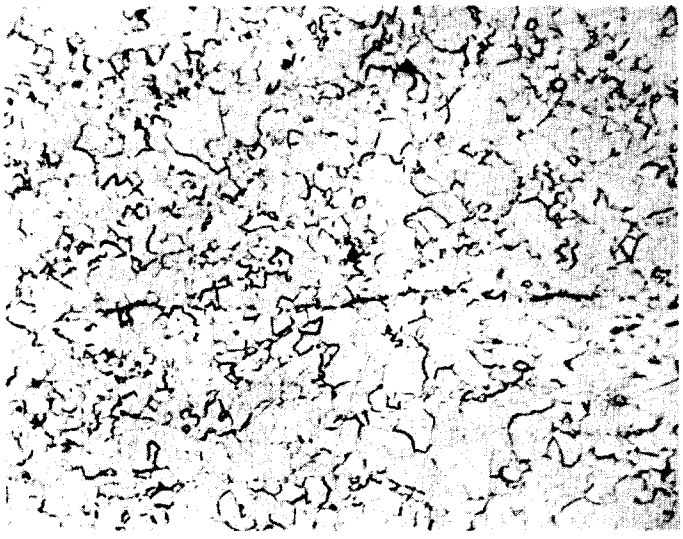
Figure 24

Neg: 230 Mag: 500X
Spec: M657-4 Etch: Phospho-Picral, 15 sec

Longitudinal section from 742 Ha(H), high temperature annealed 1 hour at 750°F. No distinct evidence of the finely divided phase was observed but there was no significant change in the more massive phase.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 13.3 | 17.4 | 30.7 | 15 |

Figure 25

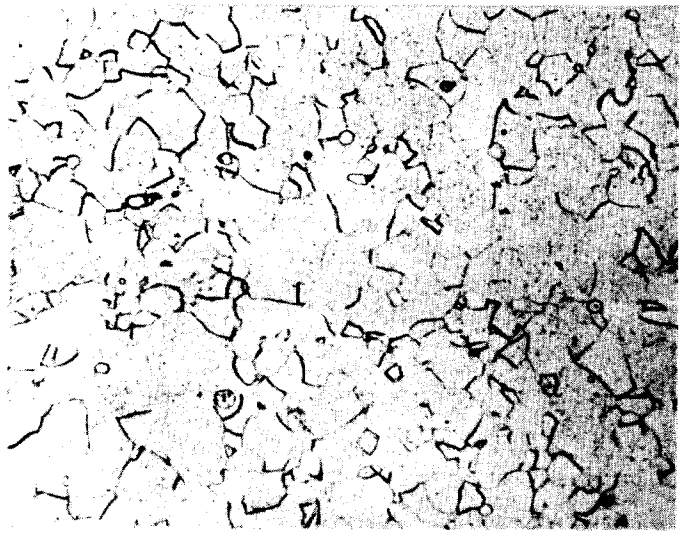


Neg: 234 Mag: 500X
Spec: M655-4 Etch: Phospho-Picral and Acetic Picral

Longitudinal microstructure of R-742Hs (magnesium-0.76 zinc-0.31 cerium, 0.41 cerium/zinc ratio), but rolled and heated 1 hour at 600°F. The degree of recrystallization may not have been entirely complete and the grain size was extremely fine, considerably less than 0.00022".

| CYS | TYS | UTS | %El. |
|---|---|---|---|
| 18.3 | 27.7 | 37.0 | 22 |

Figure 26

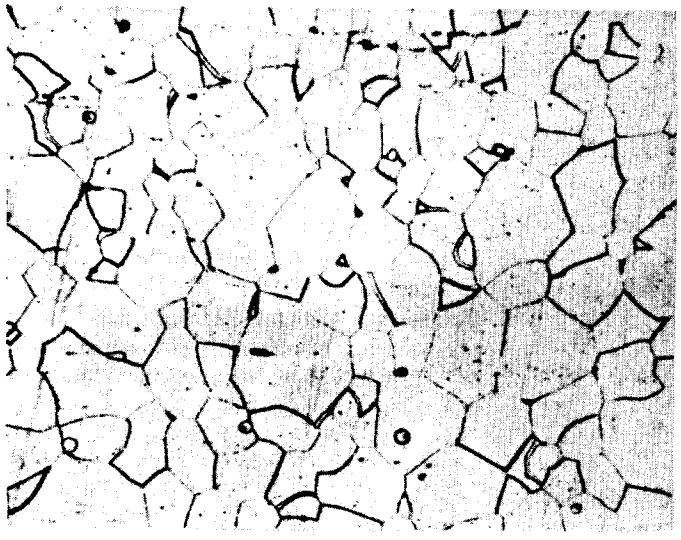


Neg: 235 Mag: 500X
Spec: M657-2 Etch: Same as Fig. 25

Longitudinal structure of R-742Ha(L), hot rolled and heated 1 hour at 650°F. The structure appeared completely recrystallized, equi-axed, with an average grain size of 0.00031". Compare with Fig. 23.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 15.6 | 21.0 | 33.5 | 15 |

Figure 27



Neg: 236 Mag: 500X
Spec: M657-4 Etch: Same as Fig. 25

Longitudinal section of R-742Ha(H), hot rolled and heated 1 hour at 750°F. Grain growth had occurred and the average grain size was 0.00062". Compare with Fig. 24 and note what appears to be undissolved phase along some grain boundaries.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 13.3 | 17.4 | 30.7 | 15 |

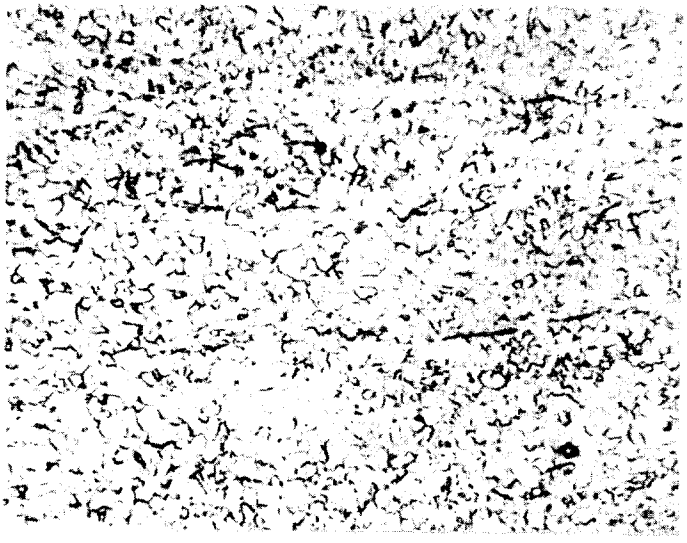


Figure 28

Neg: 231 Mag: 500X
Spec: M656-4 Etch: Same as Fig. 25

Longitudinal section of R-742Ws (magnesium-0.76 zinc-0.31 cerium, 0.41 cerium/zinc ratio) warm rolled and stress relieved 1 hour at 550°F. The degree of recrystallization did not appear complete and the grain structure was extremely fine. Compare with Fig. 25.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 17.9 | 27.3 | 36.3 | 10 |

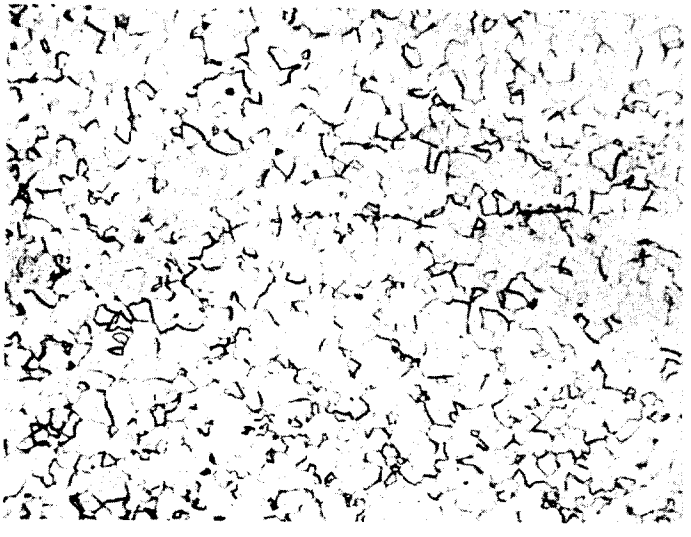


Figure 29

Neg: 232 Mag: 500X
Spec: M658-2 Etch: Same as Fig. 25

Longitudinal section of R-742Wa(L), warm rolled and heated 1 hour at 600°F. The grain structure was extremely fine averaging 0.00022".

| CYS | TYS | UTS | %El. |
|---|---|---|---|
| 14.9 | 25.2 | 34.7 | 15 |

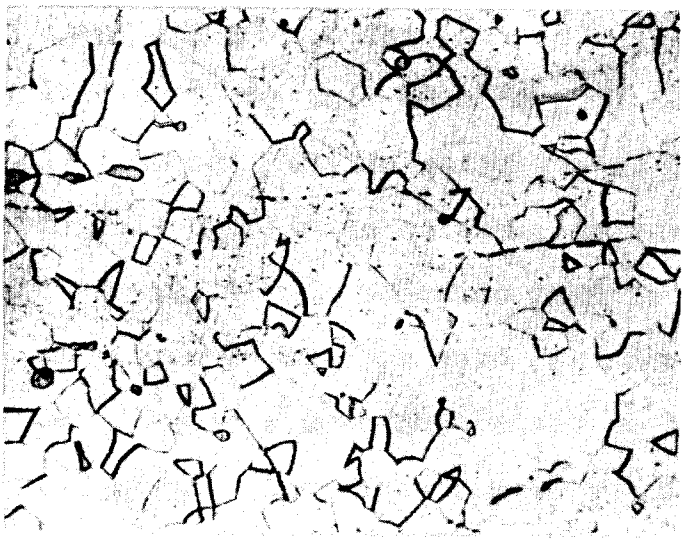


Figure 30

Neg: 233 Mag: 500X
Spec: M658-4 Etch: Same as Fig. 25

Longitudinal section of R-742Wa(H), warm rolled and heated 1 hour at 750°F. The grain size averaged 0.00042". Compare with Fig. 27.

| CYS | TYS | UTS | % El. |
|---|---|---|---|
| 11.5 | 20.6 | 32.6 | 14 |

tures. The appearance of the condition is illustrated in Figs. 24, 27 and 30 for hot and warm rolled sheet. Observations indicated that the grain boundary effect was definitely associated with higher cerium analyses. Suspecting that the condition was evidence of grain boundary melting, additional samples of R-742H and 745H sheet (0.41 and 0.64 cerium/zinc ratios) were heated from 700°F to 900°F in steps of 50°F to determine if the solidus could be detected. Microscopic examination indicated no distinct evidence of grain boundary melting up to 900°F. No conclusive evidence was obtained to explain the appearance of the white phase along grain boundaries.

The results of this investigation of small variations in zinc and cerium are summarized below:

1. The optimum hot rolling temperature for the investigation was 700°F. Warm rolling was satisfactory at 400°F.
2. For warm rolled sheet, maximum strength properties and maximum elongation values were obtained at approximately the same cerium to zinc ratio. In the range of composition studied (0.6 to 0.8% zinc and 0 to 0.5% cerium, nominally) warm rolling offered the most flexible method of sheet preparation since high elongations and a combination of high strength properties and high elongations were obtained over a range of cerium to zinc ratios (0.325 to 0.425) and over a range of heat treatment temperatures from stress relieving to low

temperature annealing conditions.

3. Although the attractive mechanical properties of the warm rolling procedures were duplicated by one set of hot rolling conditions - a cerium/zinc ratio of 0.41 and a stress relieving heat treatment - hot rolling technique was less desirable for these reasons:
   a. The cerium/zinc ratio for maximum elongation was extremely critical. Values dropped rapidly on either side of the optimum ratio, 0.41. Low temperature annealing resulted in a significant decrease in this property.
   b, The cerium/zinc ratio for maximum strength properties (approximately 0.6) was considerably above the ratio for maximum elongation (0.41).
   c. An undesirable scatter in strength properties occurred to a much greater extent than encountered in warm rolling for a given set of conditions.
4. The strength properties in hot and warm rolled sheet were approximately the same at the optimum cerium/zinc range for maximum elongation.
5. High temperature annealing practices (at the upper limit of the recrystallization temperature range) resulted in a consistent lowering of both strength and elongation values compared to lower temperatures of thermal treatments for both warm and hot rolled sheet.
6. The desirable combination of high strength properties

and high elongation values was associated with an extremely fine grain size and a relatively small amount of undissolved secondary phase.

7. The finest grain structures were associated with higher cerium analyses and with the stress relieved condition for warm and hot rolled sheet.

8. The extent of the undissolved secondary phase (or phases) ranged from less than 5% of the structure to greater than 40% with cerium analyses from 0.21% to 0.47%, respectively. The distribution of the phase apparently was independent of the method of sheet working but was influenced considerably by the temperature of heat treatment. The solid solubility of the phase was believed to be less than 0.31% cerium in combination with nominally 0.8 zinc at 750°F.

Plans for an Extension of Study of the Magnesium-Zinc-Cerium System

Plans for additional work with magnesium-zinc-cerium alloys were made from the indications given by the study of the effects of small variations in zinc and cerium on mechanical properties. Three separate phases of work were planned as outlined below:

1. McDonald[1,2] demonstrated in investigations of magnesium-base binary alloys that the optimum combination of strength properties and maximum elongation values was obtained with a magnesium-3.0 zinc alloy and a

magnesium-0.3 cerium alloy. Therefore, it was planned to extend the magnesium-zinc-cerium investigation to alloys containing more than 0.8% zinc in combinations with 0.3 cerium. A second phase of the problem was to determine whether or not the optimum cerium/zinc ratio established as 0.35-0.40 for the nominally magnesium-0.8 zinc-0.3 cerium alloy is a fundamental parameter that may be used with a higher range of zinc and cerium additions.

2. New heats intended to approximate R-742 (magnesium-0.76 zinc-0.31 cerium, cerium to zinc ratio, 0.407) were planned in order to investigate the following conditions for warm and hot rolled sheet:
   a. Study more completely the entire recrystallization temperature range.
   b. Determine notch sensitivity properties.
   c. Determine minimum bend radii.
3. During the course of the investigation of small variations in zinc and cerium, a preliminary study was made to determine if the cold rolling of hot rolled sheet prior to heat treatment would substitute for the warm rolling procedure. This work was confined to a single composition (magnesium-0.8 zinc-0.2 cerium, to simulate R-613 and R-669) and one range of cold work (nominally 15%). The results obtained were not encouraging and the study was abandoned temporarily. It was planned to

resume this work to investigate more completely a range of cold work and a range of heat treatment temperatures.

Investigation of Large Variations in Zinc and Cerium

A new series of 11 compositions was prepared (a) to investigate the effect of increasing the zinc additions from 0.8 to 3.0% with a nominal cerium addition of 0.3%, and (b) to investigate the effect of increasing the zinc and cerium additions simultaneously to maintain a constant ratio of cerium to zinc of 0.4. Intended compositions of these alloys are listed in Table XIII.

TABLE XIII

INTENDED COMPOSITIONS OF MAGNESIUM-ZINC-CERIUM ALLOYS TO INVESTIGATE LARGER VARIATIONS IN ZINC AND CERIUM

| Intended Weight Zinc | Composition Percent Cerium | Ratio Cerium/Zinc |
|---|---|---|
| 0.8 | 0.3 | 0.4 |
| 1.0 | 0.3 | 0.3 |
| 1.5 | 0.3 | 0.2 |
| 2.0 | 0.3 | 0.15 |
| 2.5 | 0.3 | 0.12 |
| 3.0 | 0.3 | 0.10 |
| 1.0 | 0.4 | 0.4 |
| 1.5 | 0.6 | 0.4 |
| 2.0 | 0.8 | 0.4 |
| 2.5 | 1.0 | 0.4 |
| 3.0 | 1.2 | 0.4 |

Difficulty was encountered in obtaining the higher cerium concentrations proposed in Table XIII. In particular, the alloys containing a cerium to zinc ratio of 0.4 with higher ranges of zinc were not obtained consistently. These off-composition heats, however, were processed along with the others for the information that they would provide.

A list of mechanical properties not reported previously for alloys in this investigation is given in Table XIV. A summary of average mechanical properties of all alloys in the investigation of larger variations in zinc and cerium additions is given in Table XV. In order to assist the interpretation of the information presented in Table XV, the elongation values have been plotted on coordinates of cerium and zinc spectrographic analyses in Figs. 31, 32 and 33. In Fig. 31, the maximum values of elongation for each alloy in the warm rolled and heat treated condition are shown. The maximum values of elongation for each alloy in the hot rolled and heat treated condition are given in Fig. 32. The distribution of maximum values of elongation for all alloys in all conditions are shown in Fig. 33.

The results of this investigation indicate that there is a distinct range or region of composition in which elongations of 20% in 2 in. or greater combined with relatively high strength properties can be obtained. As predicted in Fig. 33, this region extends from approximately 0.7 to 1.5% zinc and from approximately 0.25 to at least 0.4% cerium. Although elongations exceeding 20% in 2 inches were obtained with cerium concentrations below 0.2%,

TABLE XIV

MECHANICAL PROPERTIES OF MAGNESIUM-ZINC-CERIUM ALLOYS

TO INVESTIGATE LARGER VARIATIONS IN ZINC AND CERIUM

| Alloy | Composition Intended and (Spectro. Anal.) Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|---|
| 778Ws | 1.6 | 0.13 | 700 | 400 | 1 | 450 | - | 29.9 | 38.4 | 8.0 |
| | | | | | | | 24.9 | 31.5 | 38.7 | 11.5 |
| | | | | | | | 24.5 | 28.9 | 38.7 | 11.0 |
| | | | | | | (average) | 24.7 | 30.1 | 38.6 | 10.2 |
| 778Wa(L) | - | - | 700 | 400 | 1 | 575 | 14.1 | 25.2 | 35.1 | 16.0* |
| | | | | | | | 15.5 | 26.2 | 34.8 | 22.5 |
| | | | | | | | 15.1 | 25.5 | 33.8 | 19.5 |
| | | | | | | (average) | 14.9 | 25.6 | 34.6 | 20.0 |
| 781Hs | 1.0 | 0.10 | 700 | - | 1 | 500 | 23.5 | 27.8 | 36.5 | 15.0* |
| | | | | | | | 23.6 | 31.6 | 37.8 | 11.0 |
| | | | | | | | 24.3 | 32.2 | 38.1 | 11.5 |
| | | | | | | (average) | 23.8 | 30.5 | 37.5 | 12.5 |
| 781Ha(L) | - | - | 700 | - | 1 | 600 | 17.4 | 25.2 | 34.6 | 18.5 |
| | | | | | | | 18.0 | 26.4 | 35.7 | 12.5* |
| | | | | | | | 18.7 | 29.6 | 35.9 | 15.5 |
| | | | | | | (average) | 18.0 | 27.1 | 35.4 | 17.0 |
| 781Ws | - | - | 700 | 400 | 1 | 500 | 17.8 | 28.2 | 36.5 | 19.0* |
| | | | | | | | 17.5 | 27.8 | 36.1 | 20.5 |
| | | | | | | | 17.3 | - | 35.9 | 23.0 |
| | | | | | | (average) | 17.5 | 28.0 | 36.2 | 21.0 |
| 781Wa(L) | - | - | 700 | 400 | 1 | 600 | - | - | 34.7 | 17.5 |
| | | | | | | | 14.9 | 25.6 | 33.8 | 19.5 |
| | | | | | | | 14.5 | 25.7 | 34.7 | 18.0 |
| | | | | | | (average) | 14.7 | 25.6 | 34.4 | 18.3 |
| 788Hs | 0.91 | 0.40 | 700 | - | 1 | 450 | 22.4 | 29.4 | 36.7 | 21.0 |
| | | | | | | | 23.5 | 29.7 | 37.1 | 18.5* |
| | | | | | | | 22.3 | 30.0 | 36.7 | 10.0 |
| | | | | | | | 22.7 | 29.7 | 36.8 | 19.2 |
| 788Ha(L) | - | - | 700 | - | 1 | 475 | 22.4 | 30.3 | 37.7 | 20.0 |
| | | | | | | | 23.1 | 29.4 | 37.0 | 17.0* |
| | | | | | | | 22.7 | 30.9 | 37.3 | 21.0 |
| | | | | | | (average) | 22.7 | 30.2 | 37.3 | 20.0 |

* Specimen broke through the tensometer notch.

TABLE XIV, Cont'd

| Alloy | Composition Intended and (Spectro. Anal.) Zinc | Cerium | Nominal Rolling Temp, °F Hot | Warm | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|---|
| 788Ws | - | - | 700 | 400 | 1 | 400 | 20.5 | 27.1 | 36.6 | 22.0 |
| | | | | | | | 19.4 | 27.9 | 37.0 | 21.0* |
| | | | | | | | 19.3 | 27.2 | 36.1 | 21.0* |
| | | | | | | (average) | 19.7 | 27.4 | 36.6 | 21.5 |
| 788Wa(L) | - | - | 700 | 400 | 1 | 450 | 15.9 | 24.7 | 35.1 | 19.5 |
| | | | | | | | 15.0 | 26.1 | 35.7 | 21.0 |
| | | | | | | | 15.5 | 25.2 | 35.4 | 14.5* |
| | | | | | | (average) | 15.5 | 25.3 | 35.4 | 20.0 |
| 789Hs | 1.0 | 0.27 | 700 | - | 1 | 325 | 16.7 | 27.0 | 34.9 | 19.5 |
| | | | | | | | 17.4 | 28.2 | 34.9 | 15.5 |
| | | | | | | | 17.0 | 27.2 | 34.7 | 20.0 |
| | | | | | | (average) | 17.0 | 27.5 | 34.8 | 18.5 |
| 789Ha(L) | - | - | 700 | - | 1 | 450 | 16.2 | 25.7 | 34.9 | 16.5 |
| | | | | | | | 17.1 | 25.7 | 37.2 | 17.0 |
| | | | | | | | 16.3 | 24.3 | 34.3 | 15.5 |
| | | | | | | (average) | 16.8 | 25.2 | 35.5 | 16.3 |
| 789Ws | - | - | 700 | 400 | 1 | 400 | 28.0 | 32.0 | 39.7 | 8.0 |
| | | | | | | | 27.9 | 32.2 | 39.9 | 9.0 |
| | | | | | | | 28.0 | 32.4 | 40.0 | 9.0 |
| | | | | | | (average) | 28.0 | 32.2 | 39.9 | 8.7 |
| 789Wa(L) | - | - | 700 | 400 | 1 | 550 | 19.1 | 27.0 | 35.3 | 21.5 |
| | | | | | | | 19.3 | 26.7 | 35.5 | 24.5 |
| | | | | | | | 19.9 | 27.8 | 35.7 | 13.0* |
| | | | | | | (average) | 19.4 | 27.3 | 35.5 | 23.0 |
| 791Hs | 0.90 | 0.15 | 700 | - | 1 | 600 | 17.8 | 26.2 | 33.5 | 8.0 |
| | | | | | | | 17.8 | 29.4 | 36.7 | 20.0 |
| | | | | | | | 17.5 | 29.3 | 36.8 | 12.0 |
| | | | | | | (average) | 17.7 | 28.3 | 35.7 | 13.5 |
| 791Ha(L) | - | - | 700 | - | 1 | 675 | 26.0 | 33.6 | 38.2 | 18.0 |
| | | | | | | | 24.4 | 28.3 | 35.4 | 16.5 |
| | | | | | | | 23.9 | 28.5 | 35.3 | 17.5 |
| | | | | | | (average) | 24.7 | 30.1 | 36.3 | 17.3 |
| 791Ws | - | - | 700 | 400 | 1 | 450 | 28.6 | 30.6 | 36.2 | 15.5 |
| | | | | | | | 27.3 | 32.0 | 39.3 | 9.0 |
| | | | | | | | 28.6 | 31.9 | 39.3 | 8.0 |
| | | | | | | (average) | 28.2 | 31.5 | 38.3 | 10.5 |

TABLE XIV, Cont'd

| Alloy | Composition Intended and (Spectro. Anal.) Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|---|
| 791Wa(L) | - | - | 700 | 400 | 1 | 550 | 21.4 | 29.9 | 36.5 | 16.5 |
| | | | | | | | 20.8 | 29.4 | 36.7 | 21.0 |
| | | | | | | | 21.7 | 30.3 | 36.8 | 16.5* |
| | | | | | | (average) | 21.3 | 29.9 | 36.7 | 18.0 |
| 793Hs | 0.80 | 0.15 | 700 | - | 1 | 450 | 18.7 | 25.6 | 33.4 | 9.5 |
| | | | | | | | 20.3 | 22.4 | 29.2 | 16.0 |
| | | | | | | | 18.9 | 27.1 | 34.2 | 9.5 |
| | | | | | | (average) | 19.3 | 25.0 | 32.3 | 11.7 |
| 793Ha(L) | - | - | 700 | - | 1 | 575 | 10.9 | 20.2 | 32.0 | 12.0* |
| | | | | | | | 15.8 | 20.4 | 32.6 | 20.0 |
| | | | | | | | 16.9 | 21.2 | 32.9 | 21.0 |
| | | | | | | (average) | 15.0 | 20.6 | 32.5 | 20.5 |
| 793Ws | - | - | 700 | 400 | 1 | 300 | 26.0 | 35.4 | 39.0 | 9.0 |
| | | | | | | | - | 35.9 | 39.9 | 10.5 |
| | | | | | | | - | 36.3 | 39.4 | 12.0 |
| | | | | | | (average) | 26.0 | 35.9 | 39.4 | 10.5 |
| 793Wa(L) | - | - | 700 | 400 | 1 | 575 | 15.3 | 24.5 | 33.7 | 18.5 |
| | | | | | | | 14.7 | 25.9 | 35.2 | 20.0 |
| | | | | | | | 13.4 | 24.7 | 34.2 | 21.5 |
| | | | | | | (average) | 14.5 | 25.0 | 34.4 | 20.0 |
| 794Hs | 1.4 | 0.23 | 700 | - | 1 | 575 | 18.3 | 24.5 | 34.6 | 18.0 |
| | | | | | | | 18.2 | 24.2 | 34.8 | 14.0* |
| | | | | | | | 18.1 | - | 37.6 | 15.0* |
| | | | | | | (average) | 18.2 | 24.3 | 35.7 | 15.8 |
| 794Ha(L) | - | - | 700 | - | 1 | 700 | 13.0 | 15.8 | 29.6 | 20.0 |
| | | | | | | | 13.1 | 20.2 | 33.4 | 16.5* |
| | | | | | | | 13.4 | 17.9 | 32.9 | 9.0* |
| | | | | | | (average) | 13.2 | 18.0 | 32.0 | 17.0 |
| 794Ws | - | - | 700 | 400 | 1 | 350 | 29.9 | 35.8 | 44.2 | 13.0 |
| | | | | | | | 30.4 | 35.0 | 42.4 | 11.5 |
| | | | | | | | 30.0 | 34.0 | 41.6 | 9.5 |
| | | | | | | (average) | 30.1 | 34.9 | 42.8 | 11.3 |
| 794Wa(L) | - | - | 700 | 400 | 1 | 500 | 22.1 | 30.2 | 38.2 | 18.0 |
| | | | | | | | 21.8 | 32.0 | 39.4 | 12.0* |
| | | | | | | | 22.2 | 29.4 | 39.0 | 18.0 |
| | | | | | | (average) | 22.0 | 30.5 | 38.9 | 17.0 |

TABLE XIV, Cont'd

| Alloy | Composition Intended and (Spectro. Anal.) Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment Time | Temp. | Kips per sq.in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|---|
| 796Hs | 2.7 | 0.57 | 700 | - | 1 | 475 | 18.9 | 26.0 | 36.3 | 10.0* |
| | | | | | | | 20.0 | 26.5 | 35.6 | 8.5 |
| | | | | | | | 19.8 | 26.9 | 36.6 | 13.0 |
| | | | | | | (average) | 19.6 | 26.5 | 36.2 | 10.5 |
| 796Ha(L) | - | - | 700 | - | 1 | 550 | - | 22.0 | 34.1 | 10.0* |
| | | | | | | | 14.8 | 21.6 | 34.2 | 14.5 |
| | | | | | | | 14.5 | 21.5 | 33.9 | 14.5 |
| | | | | | | (average) | 14.6 | 21.7 | 34.1 | 14.5 |
| 796Ws | - | - | 700 | 400 | 1 | 300 | 26.4 | 34.0 | 41.1 | 6.0 |
| | | | | | | | 27.0 | 34.2 | 41.6 | 8.0* |
| | | | | | | | 27.1 | 34.6 | 41.4 | 10.0 |
| | | | | | | (average) | 26.8 | 34.3 | 41.4 | 8.0 |
| 796Wa(L) | - | - | 700 | 400 | 1 | 425 | 22.5 | 29.4 | 39.8 | 12.0 |
| | | | | | | | 21.6 | 29.8 | 39.2 | 10.5 |
| | | | | | | | 22.7 | 28.6 | 38.9 | 10.0 |
| | | | | | | (average) | 22.3 | 29.3 | 39.3 | 10.8 |
| 798Hs | 3.3 | 0.18 | 700 | - | 1 | 500 | 10.4 | 22.4 | 34.9 | 14.0 |
| | | | | | | | 10.8 | 21.7 | 24.9 | 12.5 |
| | | | | | | | 10.9 | 22.8 | 34.9 | 11.0 |
| | | | | | | (average) | 10.7 | 22.3 | 34.9 | 12.5 |
| 798Ha(L) | - | - | 700 | - | 1 | 550 | 8.9 | 22.5 | 33.6 | 11.0* |
| | | | | | | | 8.6 | 24.0 | 34.1 | 15.0* |
| | | | | | | | 8.8 | 23.6 | 33.9 | 13.0 |
| | | | | | | (average) | 8.8 | 24.3 | 33.9 | 13.0 |
| 798Ws | - | - | 700 | 400 | 1 | 350 | 15.9 | 27.2 | 37.8 | 18.5 |
| | | | | | | | 19.8 | 25.7 | 37.0 | 18.0 |
| | | | | | | | 15.7 | 28.6 | 38.8 | 18.0 |
| | | | | | | (average) | 17.1 | 27.2 | 37.9 | 18.2 |
| 798Wa(L) | - | - | 700 | 400 | 1 | 475 | 15.6 | 25.8 | 36.7 | 21.5 |
| | | | | | | | 13.3 | 24.1 | 35.7 | 17.0 |
| | | | | | | | 11.9 | 25.5 | 36.6 | 17.0 |
| | | | | | | (average) | 13.6 | 25.1 | 36.3 | 18.5 |

## TABLE XV

## SUMMARY OF AVERAGE MECHANICAL PROPERTIES OF MAGNESIUM-ZINC-CERIUM ALLOYS TO INVESTIGATE LARGER VARIATIONS IN ZINC AND CERIUM

| Alloy | Composition, Spectro. Anal. Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment One Hour at | Kips per sq. in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| 793Ws | 0.80 | 0.15 | 700 | 400 | 300°F | 26.0 | 35.9 | 39.4 | 10.5 |
| Wa(L) | - | - | 700 | 400 | 575 | 14.5 | 25.0 | 34.4 | 20.0 |
| Hs | - | - | 700 | - | 450 | 19.3 | 25.0 | 32.3 | 11.5 |
| Ha(L) | - | - | 700 | - | 575 | 15.0 | 20.6 | 32.5 | 20.5 |
| 791Ws | 0.90 | 0.15 | 700 | 400 | 450 | 28.7 | 31.5 | 38.3 | 10.5 |
| Wa(L) | - | - | 700 | 400 | 550 | 21.3 | 29.9 | 36.7 | 18.0 |
| Hs | - | - | 700 | - | 600 | 17.7 | 28.3 | 35.7 | 13.5 |
| Ha(L) | - | - | 700 | - | 675 | 24.7 | 30.0 | 36.3 | 17.5 |
| 788Ws | 0.91 | 0.40 | 700 | 400 | 400 | 19.7 | 27.4 | 36.6 | 21.5 |
| Wa(L) | - | - | 700 | 400 | 450 | 15.5 | 25.3 | 35.4 | 20.0 |
| Hs | - | - | 700 | - | 450 | 22.7 | 29.7 | 36.8 | 19.0 |
| Ha(L) | - | - | 700 | - | 575 | 22.7 | 30.2 | 37.3 | 20.0 |
| 781Ws | 1.0 | 0.10 | 700 | 400 | 500 | 17.5 | 28.0 | 36.2 | 21.0 |
| Wa(L) | - | - | 700 | 400 | 600 | 14.7 | 25.6 | 34.4 | 18.5 |
| Hs | - | - | 700 | - | 500 | 23.8 | 30.5 | 37.5 | 12.5 |
| Ha(L) | - | - | 700 | - | 600 | 18.0 | 27.1 | 35.6 | 17.0 |
| 789Ws | 1.0 | 0.27 | 700 | 400 | 400 | 28.0 | 32.2 | 39.9 | 8.5 |
| Wa(L) | - | - | 700 | 400 | 550 | 19.4 | 27.3 | 35.5 | 23.0 |
| Hs | - | - | 700 | - | 325 | 17.0 | 27.5 | 34.8 | 18.5 |
| Ha(L) | - | - | 700 | - | 450 | 16.8 | 25.2 | 35.5 | 16.5 |
| 810Ws | 1.2 | 0.31 | 700 | 400 | 350 | 29.3 | 34.9 | 42.2 | 13.0 |
| Wa(L) | - | - | 700 | 400 | 550 | 18.9 | 27.6 | 37.7 | 15.0 |
| Hs | - | - | 700 | - | 550 | 16.9 | 26.8 | 34.9 | 12.3 |
| Ha(L) | - | - | 700 | - | 625 | 16.1 | 23.6 | 34.3 | 7.5 |
| 814Ws | 1.2 | 0.38 | 700 | 400 | 450 | 24.5 | 32.6 | 40.5 | 20.5 |
| Wa(L) | - | - | 700 | 400 | 575 | 16.8 | 25.9 | 36.7 | 14.0 |
| Hs | - | - | 700 | - | 500 | 20.3 | 27.6 | 35.7 | 11.0 |
| Ha(L) | - | - | 700 | - | 650 | 12.2 | 18.4 | 31.6 | 10.0 |
| 794Ws | 1.4 | 0.23 | 700 | 400 | 350 | 30.1 | 34.9 | 42.8 | 11.5 |
| Wa(L) | - | - | 700 | 400 | 500 | 22.0 | 30.5 | 38.9 | 17.0 |
| Hs | - | - | 700 | - | 575 | 18.2 | 24.3 | 35.7 | 16.0 |
| Ha(L) | - | - | 700 | - | 700 | 13.2 | 18.0 | 32.0 | 17.0 |

TABLE XV, Cont'd

| Alloy | Composition, Spectro. Anal. Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment One Hour at | Kips per sq. in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| 807Ws | 1.4 | 0.43 | 700 | 400 | 400°F | 24.6 | 33.0 | 40.7 | 11.0 |
| Wa(L) | - | - | 700 | 400 | 525 | 14.3 | 25.0 | 35.1 | 12.0 |
| Hs | - | - | 700 | - | 325 | 18.9 | 30.7 | 38.3 | 12.0 |
| 778Ws | 1.6 | 0.13 | 700 | 400 | 450 | 24.7 | 30.1 | 38.6 | 10.0 |
| Wa(L) | - | - | 700 | 400 | 575 | 14.9 | 25.6 | 34.6 | 20.0 |
| 811Ws | 1.65 | 0.41 | 700 | 400 | 450 | 27.4 | 34.1 | 39.2 | 11.0 |
| Wa(L) | - | - | 700 | 400 | 600 | 18.0 | 25.9 | 36.8 | 11.0 |
| Hs | - | - | 700 | - | 425 | 20.5 | 32.3 | 37.3 | 9.5 |
| Ha(L) | - | - | 700 | - | 525 | 19.8 | 29.1 | 36.9 | 9.0 |
| 815Ws | 2.0 | 0.31 | 700 | 400 | 350 | 24.7 | 33.9 | 40.9 | 14.5 |
| Wa(L) | - | - | 700 | 400 | 525 | 13.1 | 25.0 | 35.8 | 14.5 |
| Hs | - | - | 700 | - | 375 | 17.9 | 27.2 | 36.4 | 17.0 |
| Ha(L) | - | - | 700 | - | 525 | 12.9 | 20.9 | 34.4 | 15.0 |
| 808Ws | 2.15 | 0.64 | 700 | 400 | 375 | 23.3 | 32.6 | 40.0 | 10.0 |
| Wa(L) | - | - | 700 | 400 | 500 | 14.8 | 25.0 | 36.6 | 17.0 |
| Hs | - | - | 700 | - | 375 | 18.9 | 29.7 | 36.3 | 13.0 |
| Ha(L) | - | - | 700 | - | 475 | 17.8 | 24.6 | 34.2 | 13.5 |
| 812Ws | 2.3 | 1.1 | 700 | 400 | 475 | 27.4 | 34.0 | 41.3 | 11.0 |
| Wa(L) | - | - | 700 | 400 | 600 | 18.2 | 27.6 | 37.8 | 9.0 |
| Hs | - | - | 700 | - | 500 | 21.5 | 20.3 | 35.7 | 11.0 |
| Ha(L) | - | - | 700 | - | 575 | 19.4 | 26.6 | 36.3 | 7.0 |
| 813Ws | 2.5 | 0.37 | 700 | 400 | 325 | 23.6 | 35.1 | 42.6 | 13.0 |
| Wa(L) | - | - | 700 | 400 | 450 | 15.4 | 26.5 | 31.2 | 12.0 |
| Hs | - | - | 700 | - | 425 | 17.5 | 26.4 | 36.1 | 13.0 |
| Ha(L) | - | - | 700 | - | 525 | 16.5 | 22.1 | 33.3 | 10.5 |
| 809Ws | 2.5 | 0.55 | 700 | 400 | 375 | 26.9 | 33.8 | 41.3 | 6.0 |
| Wa(L) | - | - | 700 | 400 | 500 | 17.4 | 25.6 | 36.7 | 13.0 |
| Hs | - | - | 700 | - | 400 | 22.2 | 30.7 | 37.0 | 12.0 |
| Ha(L) | - | - | 700 | - | 550 | 17.0 | 24.9 | 36.3 | 12.0 |
| 796Ws | 2.7 | 0.57 | 700 | 400 | 300 | 26.8 | 34.3 | 41.4 | 8.0 |
| Wa(L) | - | - | 700 | 400 | 425 | 22.3 | 29.3 | 39.3 | 11.0 |
| Hs | - | - | 700 | - | 475 | 19.6 | 26.5 | 36.2 | 10.5 |
| Ha(L) | - | - | 700 | - | 550 | 14.6 | 21.7 | 34.1 | 14.5 |

TABLE XV, Cont'd

| Alloy | Composition, Spectro. Anal. Zinc | Cerium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment One Hour at | Kips per sq. in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| 822Ws | 2.8 | 0.40 | 700 | 400 | 375°F | 16.5 | 28.2 | 38.5 | 14.5 |
| Wa(L) | - | - | 700 | 400 | 500 | 11.9 | 24.8 | 34.8 | 15.0 |
| Hs | - | - | 700 | - | 350 | 19.5 | 28.3 | 36.6 | 16.0 |
| Ha(L) | - | - | 700 | - | 450 | 14.1 | 24.1 | 35.6 | 11.0 |
| 817Ws | 3.2 | 0.31 | 700 | 400 | 325 | 17.8 | 28.5 | 37.7 | 15.0 |
| Wa(L) | - | - | 700 | 400 | 475 | 10.6 | 20.9 | 34.4 | 8.0 |
| Hs | - | - | 700 | - | 400 | 12.6 | 25.1 | 34.3 | 12.0 |
| Ha(L) | - | - | 700 | - | 525 | 8.5 | 18.0 | 32.4 | 9.0 |
| 823Ws | 3.2 | 0.34 | 700 | 400 | 375 | 17.0 | 27.2 | 37.9 | 14.0 |
| Wa(L) | - | - | 700 | 400 | 500 | 10.7 | 23.8 | 35.8 | 14.0 |
| Hs | - | - | 700 | - | 350 | 17.6 | 29.4 | 37.8 | 14.5 |
| Ha(L) | - | - | 700 | - | 475 | 10.5 | 23.0 | 34.9 | 17.0 |
| 798Ws | 3.3 | 0.18 | 700 | 400 | 350 | 17.1 | 27.2 | 37.9 | 18.0 |
| Wa(L) | - | - | 700 | 400 | 475 | 13.6 | 25.1 | 36.3 | 18.5 |
| Hs | - | - | 700 | - | 500 | 10.7 | 22.3 | 34.9 | 12.5 |
| Ha(L) | - | - | 700 | - | 550 | 8.8 | 24.3 | 33.9 | 13.0 |
| 824Ws | 3.4 | 0.39 | 700 | 400 | 325 | 18.5 | 31.1 | 39.5 | 14.0 |
| Wa(L) | - | - | 700 | 400 | 400 | 16.2 | 27.3 | 38.6 | 14.5 |
| Hs | - | - | 700 | - | 350 | 17.3 | 28.1 | 37.4 | 14.0 |
| Ha(L) | - | - | 700 | - | 500 | 11.0 | 23.0 | 34.9 | 10.5 |

these values were usually accompanied by considerably lowered strength properties (particularly compressive yield strength) compared to alloys in the favorable range with higher cerium analyses. The data in Figs. 31, 32 and 33 strongly suggest that the region of attractive mechanical properties may extend into higher zinc and cerium concentrations. These data also suggest that compositions falling along the nominal cerium to zinc ratio of 0.4 are centrally located in this zone. Unfortunately, alloys intended to explore this region of higher zinc and cerium concentrations were found to be much lower in cerium than expected.

The best combinations of mechanical properties in this favorable region were exhibited by the following alloys and conditions:

| Alloy | Spect. Anal. Zinc | Spect. Anal. Cerium | Heated 1 Hour at | Kips per sq. in. CYS | Kips per sq. in. TYS | Kips per sq. in. UTS | Elong. % in 2 in. |
|---|---|---|---|---|---|---|---|
| 814Ws | 1.2 | 0.38 | 450°F | 24.5 | 32.6 | 40.5 | 20.5 |
| 788Ws | 0.91 | 0.40 | 400 | 19.7 | 27.4 | 36.6 | 21.5 |
| Wa(L) | | | 450 | 15.5 | 25.3 | 35.4 | 20.0 |
| Hs | | | 450 | 22.7 | 29.7 | 36.8 | 19.0 |
| Ha(L) | | | 575 | 22.7 | 30.2 | 37.3 | 20.0 |

The properties of alloy R-788 are cited in particular because of the apparent independence of the method of sheet preparation.

Alloys with zinc concentrations approximately 1.5 to 3.0% showed no significant increase in strength properties and a generally lower level of elongation values than observed for the favorable region.

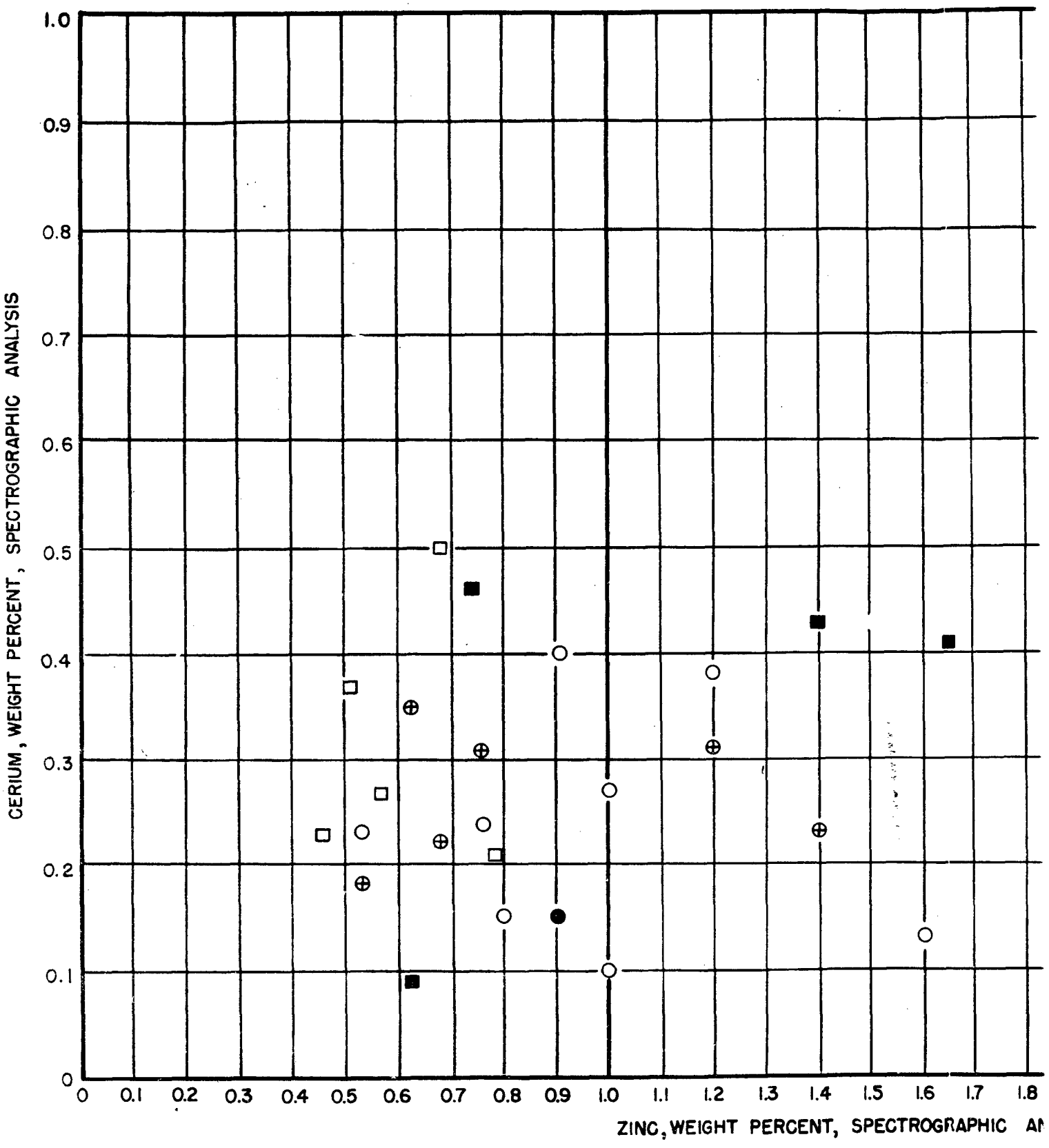
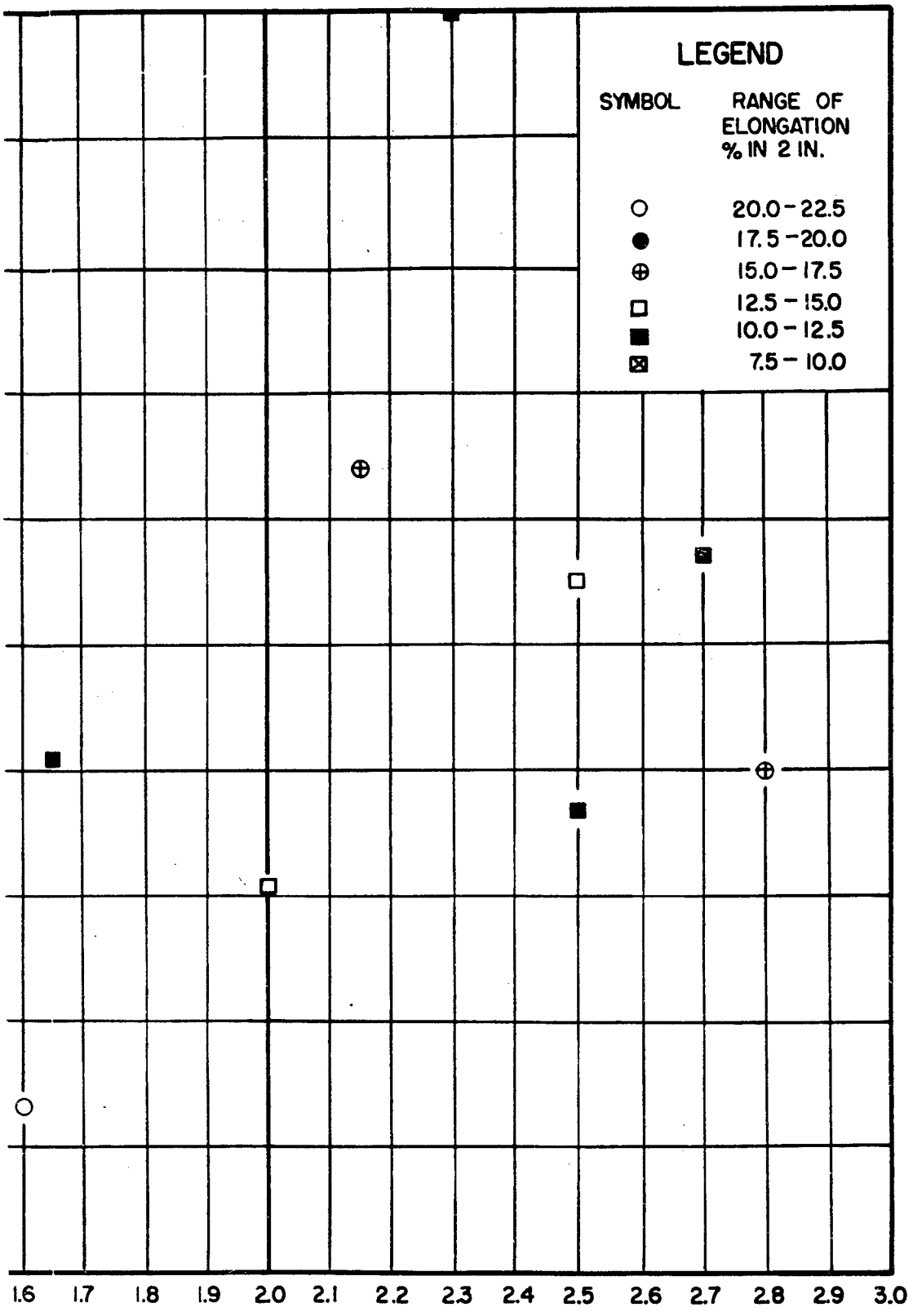



FIGURE 31

DISTRIBUTION OF THE MAXIMUM VALUES OF ELONGA
FOR ALLOYS IN THE SYSTEM MAGNESIUM-ZINC-CE
ROLLED AND HEAT TREATED CONDITION.




ROGRAPHIC ANALYSIS

31

OF ELONGATION, PERCENT IN 2 IN.,
IM-ZINC-CERIUM IN THE WARM

1.

(2)

9

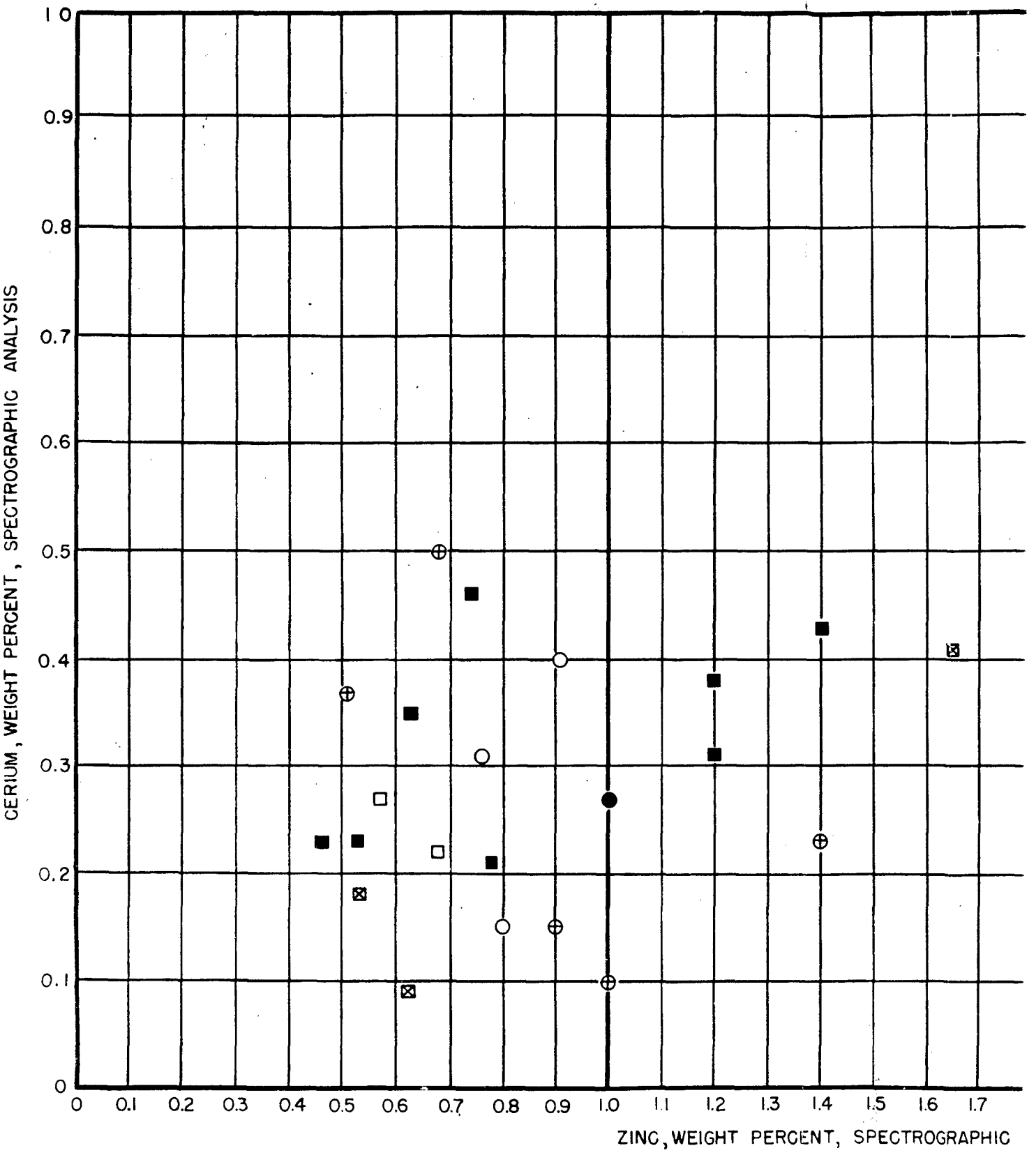
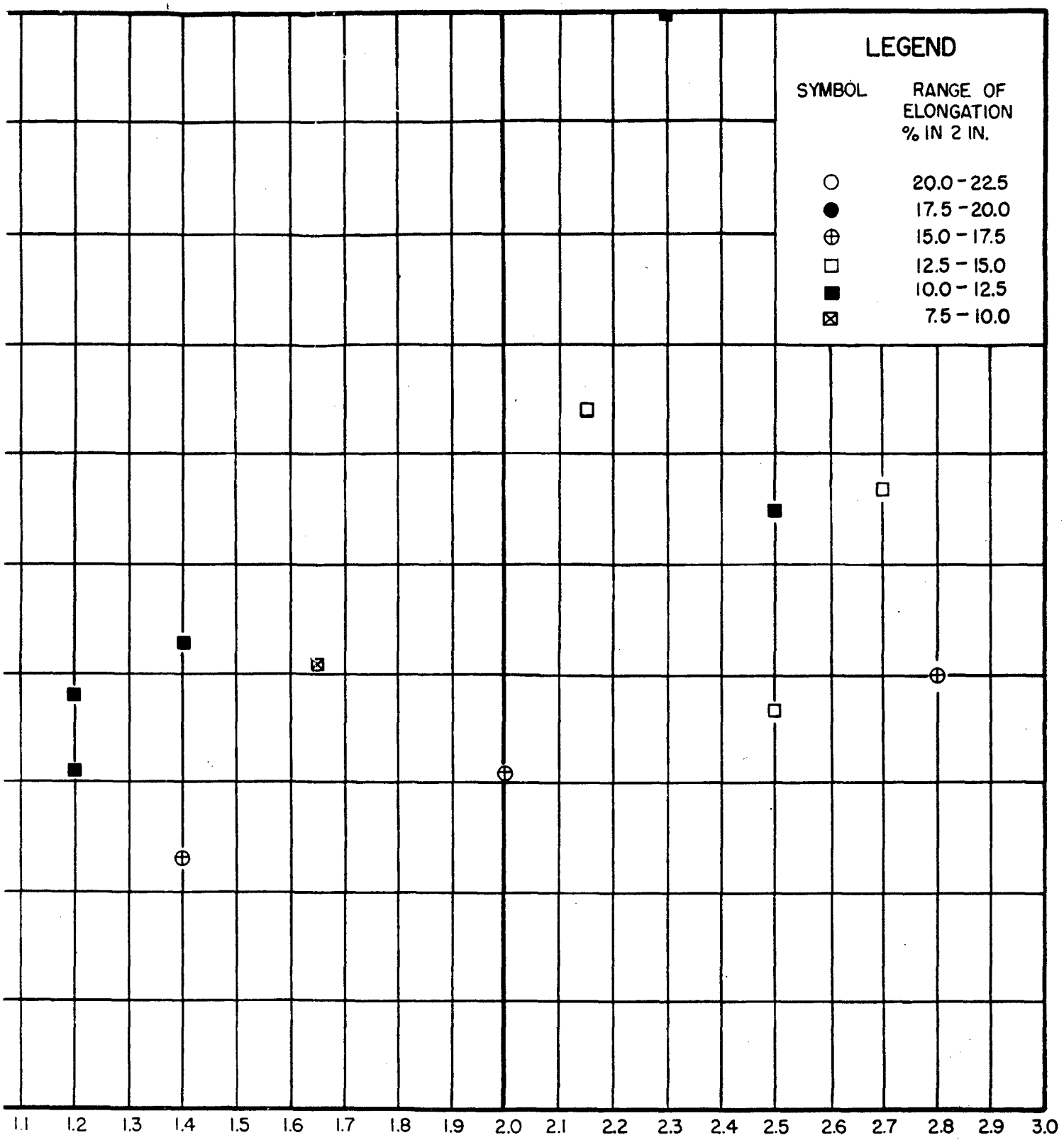



FIGURE 32

DISTRIBUTION OF THE MAXIMUM VALUES OF ELONG
FOR ALLOYS IN THE SYSTEM MAGNESIUM-ZINC-
ROLLED AND HEAT TREATED CONDITION.




IC, WEIGHT PERCENT, SPECTROGRAPHIC ANALYSIS

FIGURE 32

THE MAXIMUM VALUES OF ELONGATION, PERCENT IN 2 IN.,
HE SYSTEM MAGNESIUM-ZINC-CERIUM IN THE HOT
TREATED CONDITION.

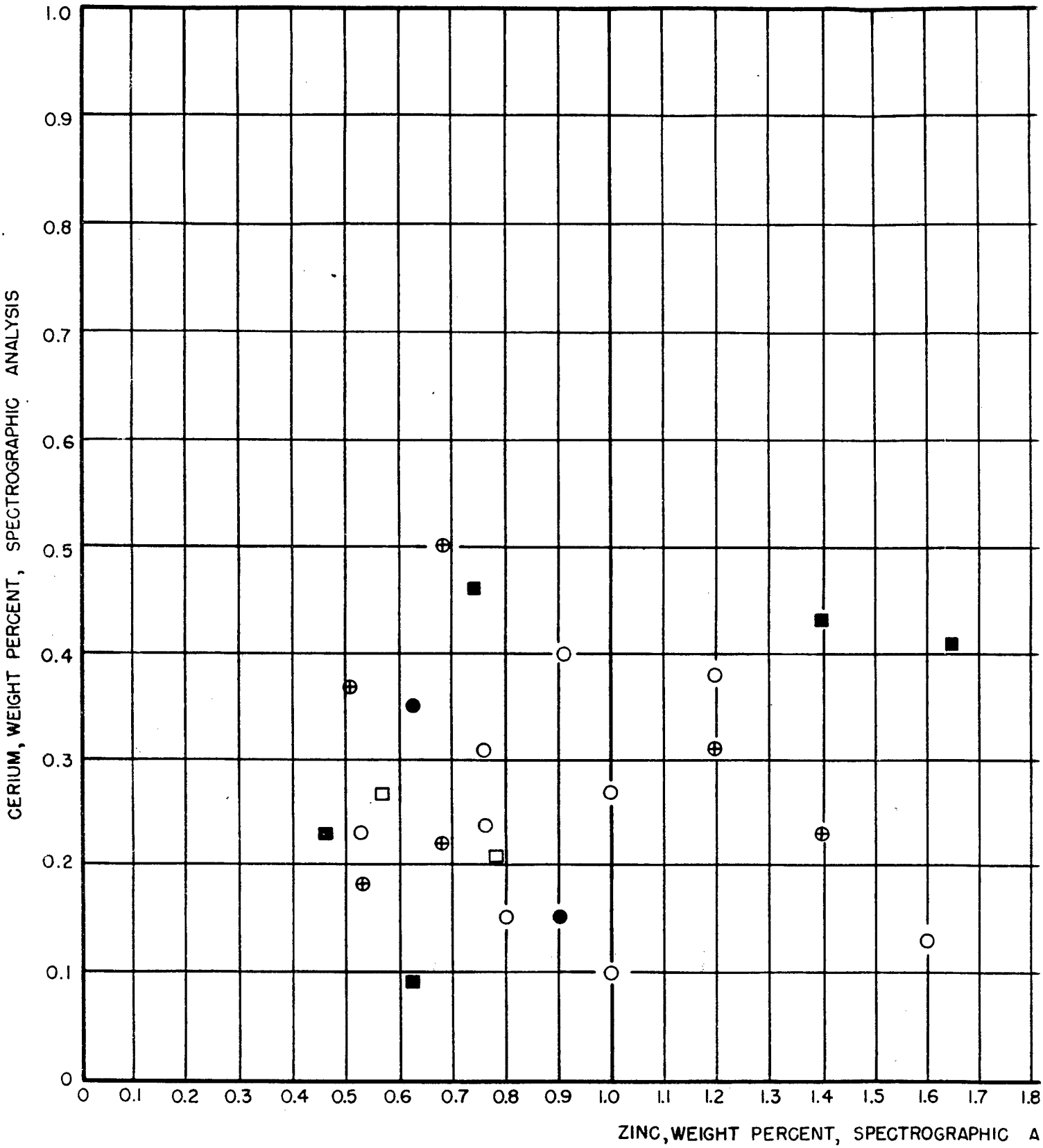



FIGURE 33

DISTRIBUTION OF THE MAXIMUM VALUES OF ELONGA
FOR ALLOYS IN THE SYSTEM MAGNESIUM-ZINC-CE

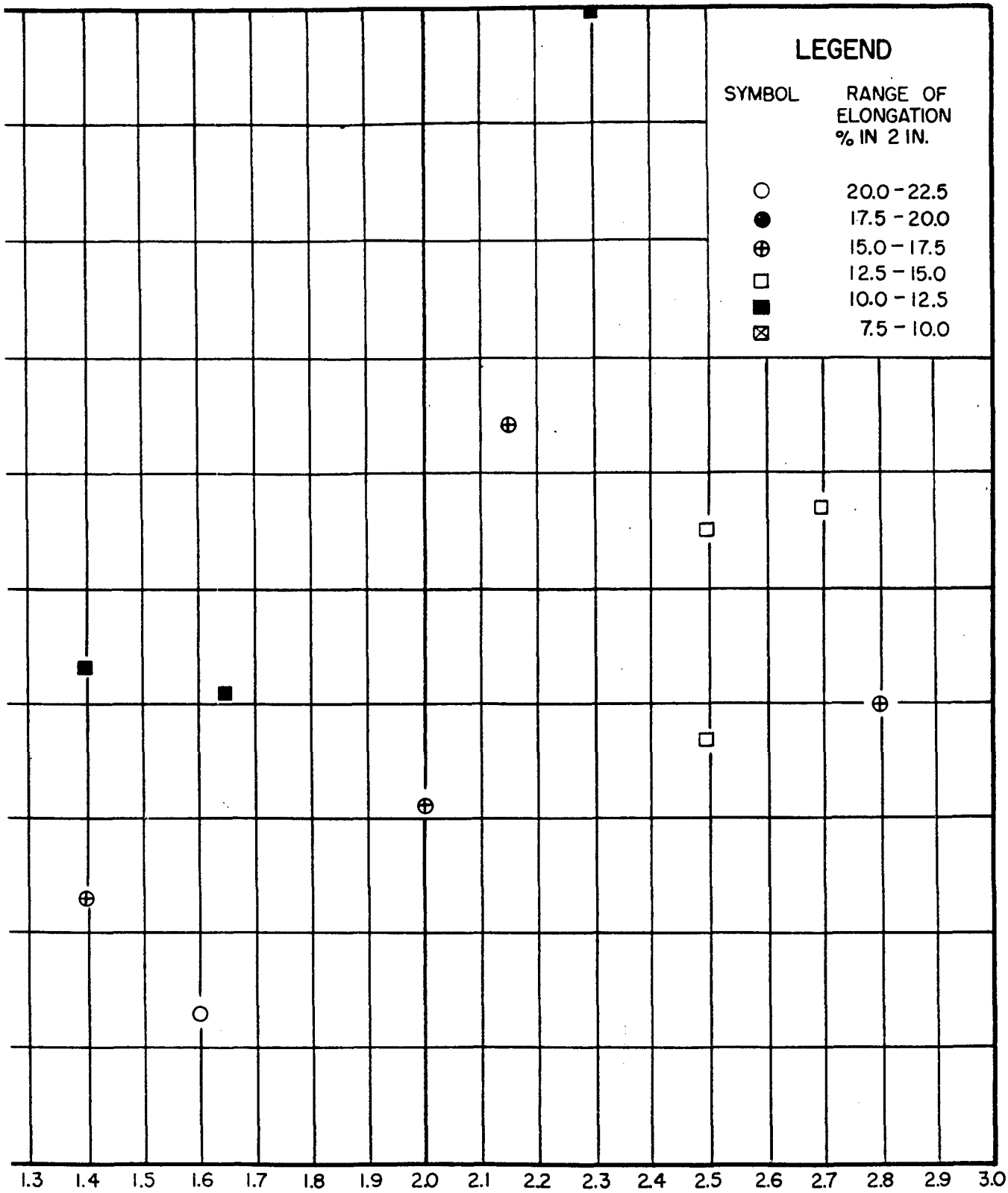



FIGURE 33

MUM VALUES OF ELONGATION, PERCENT IN 2IN.,
EM MAGNESIUM-ZINC-CERIUM.

The most significant indications from the results of the study of larger variations in zinc and cerium additions are summarized below.

1. A favorable region of composition extending from approximately 0.7 to 1.5% zinc and from 0.25 to at least 0.4% cerium was located in which a combination of high elongations and relatively high strength properties were obtained.
2. The mechanical property data predicted that the favorable region may extend to higher concentrations of zinc and cerium.
3. Zinc additions in the range from 1.5 to 3.0 in combination with 0.3 cerium or a greater cerium concentration provided no sensible increase in strength properties and resulted in consistently lower elongation values.
4. The importance of the cerium to zinc ratio of 0.4 as a prediction of favorable mechanical properties in regions of higher zinc and cerium concentrations was not established. Additional alloys along this ratio of zinc and cerium additions will be needed to establish this relation conclusively. The evidence obtained, however, indicated strongly that the 0.4 ratio was approximately in the center of the favorable region.
5. With few exceptions, the method of warm rolling produced the best combination of strength properties and elongation values.

Detailed Investigation of the Most Favorable Composition

Initially, it was planned to study more completely the method of sheet preparation and to determine notch sensitivity properties and minimum bend radii for alloy R-742 (magnesium-0.76 zinc-0.31 cerium). This experimental work was not carried out in order to complete the larger program of surveying the mechanical properties of alloys with larger concentrations of zinc and cerium. On the basis of these results it is now believed that the alloy for the detailed study should contain larger amounts of zinc and cerium. The most suitable composition for this purpose is believed to be R-788 (magnesium-0.91 zinc-0.40 cerium). The work on the R-742 type alloy already started will be completed and accompanied by the additional work on the R-788 type alloy.

Investigation of Cold Rolling and Heat Treatment to Substitute for Warm Rolling Procedure

Experimental work on this problem was not started. It is planned to begin the study after the most favorable range of zinc and cerium is established.

Comparison of Mechanical Properties of Experimental and Commercial Alloys

The results of this investigation have shown that the relatively high elongation values and high combination of strength properties exhibited by R-613Ws and R-669Ws (magnesium-0.8 zinc-0.2 cerium) were not only reproduced but also improved. This is shown in Table XVI together with a comparison between the experimental magnesium-zinc-cerium alloys and commercial alloys.

## TABLE XVI

## COMPARISON OF MECHANICAL PROPERTIES OF EXPERIMENTAL MAGNESIUM-ZINC-CERIUM AND COMMERCIAL SHEET ALLOYS

| Alloy | Composition, Nominal and (Spectro. Anal.) | Condition | Kips per sq.in. CYS | TYS Typ. | TYS Min. | UTS Typ. | UTS Min. | Elong. % 2 in. Typ. | Elong. % 2 in. Min. |
|---|---|---|---|---|---|---|---|---|---|
| Fs-1a | Mg-3 Al-1 Zn | Annealed | 16.0 | 22.0 | 15.0 | 37.0 | 32.0 | 18 | 12 |
| Fs-1h | | Hard Rolled | 26.0 | 33.0 | 26.0 | 43.0 | 38.0 | 11 | 4 |
| Ma | Mg-1.2 Mn | Annealed | 12.0 | 15.0 | 12.0 | 33.0 | 28.0 | 17 | 12 |
| Mh | | Hard Rolled | 20.0 | 29.0 | 22.0 | 37.0 | 32.0 | 8 | 4 |
| J-1a | Mg-6 Al-1 Zn | Annealed | 16.0 | 26.0 | 19.0 | 43.0 | 37.0 | 16 | 8 |
| J-1h | | Hard Rolled | 27.0 | 34.0 | 32.0 | 47.0 | 42.0 | 9 | 3 |
| Electron AM537 | Mg-2 Mn-.5 Ce | Not specified | - | 25.4 | 24.0 | 32.4 | 31.8 | 21 | 18 |
| R-613Ws | Mg-0.8 Zn-0.2 Ce | Warm Rolled at 400°F, stress relieved at 600°F | 16.4 | 25.3 | - | 35.0 | - | 23 | - |
| R-669Ws | Mg-0.8 Zn-0.2 Ce (0.76) (0.24) | Same as R-613Ws | 14.1 | 23.8 | - | 34.3 | - | 20 | - |
| R-756Ws | Mg-0.8 Zn-0.2 Ce (0.68) (0.22) | Warm Rolled at 400°F, stress relieved at 450°F | 24.1 | 31.3 | - | 39.1 | - | 16 | - |
| R-613Wa | - | Annealed at 750°F | 11.5 | 21.5 | - | 33.2 | - | 19 | - |
| R-669Wa | - | Same as R-613Wa | 14.1 | 23.8 | - | 34.3 | - | 20 | - |
| R-747Wa (L) | Mg-0.8 Zn-0.2 Ce (0.54) (0.23) | Warm Rolled at 400°F, annealed at 600°F | 16.9 | 26.1 | - | 35.2 | - | 20 | - |

TABLE XVI, Cont'd

| Alloy | Composition, Nominal and (Spectro. Anal.) | Condition | Kips per sq.in. CYS | TYS Typ. | TYS Min. | UTS Typ. | UTS Min. | Elong. % 2 in. Typ. | Elong. % 2 in. Min. |
|---|---|---|---|---|---|---|---|---|---|
| R-742Hs | Mg-0.8 Zn-0.3 Ce (0.76) (0.31) | Hot Rolled at 700°F, stress relieved at 600°F | 18.3 | 28.3 | - | 37.1 | - | 21 | - |
| R-793Ha(L) | Mg-(0.8)Zn-(0.15) Ce | Hot Rolled at 700°F, annealed at 575°F | 15.0 | 20.6 | - | 32.5 | - | 20.5 | - |
| R-791Wa(L) | Mg-(0.9)Zn-(0.15) Ce | Warm Rolled at 400°F, annealed at 550°F | 21.3 | 29.9 | - | 36.7 | - | 18.0 | - |
| R-791Ha(L) | - | Hot Rolled at 700°F, annealed at 675°F | 24.7 | 30.1 | - | 36.3 | - | 17.5 | - |
| R-788Ws | Mg-(0.91)Zn-(0.40)Ce | Warm Rolled at 400°F, stress relieved at 400°F | 19.7 | 27.4 | - | 36.6 | - | 21.5 | - |
| R-788Wa(L) | - | Warm Rolled at 400°F, annealed at 400°F | 15.5 | 25.3 | - | 35.4 | - | 20.0 | - |
| R-788Hs | - | Hot Rolled at 700°F, stress relieved at 450°F | 22.7 | 29.7 | - | 36.8 | - | 19.0 | - |
| R-788Ha(L) | - | Hot Rolled at 700°F, annealed at 475°F | 22.7 | 36.2 | - | 37.3 | - | 20.0 | - |
| R-781Ws | Mg-(1.0)Zn-0.10 Ce | Warm Rolled at 400°F, stress relieved at 500°F | 17.5 | 28.0 | - | 36.2 | - | 21.0 | - |

TABLE XVI, Cont'd

| Alloy | Composition, Nominal and (Spectro. Anal.) | Condition | Kips per sq.in. CYS | TYS Typ. | TYS Min. | UTS Typ. | UTS Min. | Elong. % 2 in. Typ. | Elong. % 2 in. Min. |
|---|---|---|---|---|---|---|---|---|---|
| R-789Wa(L) | Mg-(1.0)Zn-0.27 Ce | Warm Rolled at 400°F, annealed at 550°F | 19.4 | 27.3 | - | 35.5 | - | 23.0 | - |
| R-814Ws | Mg-1.2 Zn-0.38 Ce | Warm Rolled at 400°F, stress relieved at 450°F | 24.5 | 32.6 | - | 40.5 | - | 20.5 | - |
| R-778Wa(L) | Mg-1.6 Zn-0.13 Ce | Warm Rolled at 400°F, annealed at 575°F | 14.9 | 25.6 | - | 34.6 | - | 20.0 | - |

## Future Work with Magnesium-Zinc-Cerium Alloys

Favorable results with some alloys in this system indicate the desirability of continuing the mechanical property survey. The ultimate end of this work would be to establish a composition range and method of sheet fabrication for an optimum combination of strength and formability.

Experimental work in progress or planned includes the following:

1. Prepare new alloys having a nominal cerium to zinc ratio of 0.4 with the zinc concentrations above 1.0%. The purpose of these compositions would be to investigate the validity of the 0.4 ratio as a parameter in predicting an optimum combination of strength and ductility properties.
2. Prepare new alloys to survey the range of composition between 0.8 to 1.5% zinc and 0.4 to 0.6% cerium. This work would determine if the region of favorable compositions could be extended to higher zinc and cerium concentrations.
3. Investigate in more detail for the entire recrystallization range, notch sensitivity properties and minimum bend radii of two or more alloys exhibiting attractive mechanical properties. Two alloys selected are R-742 (magnesium-0.76 zinc-0.31 cerium) and R-788 (magnesium-0.9 zinc-0.40 cerium).
4. Investigate the possibility of substituting some com-

bination of cold rolling and heat treatment for the warm rolling procedure in sheet preparation.

5. Investigate the effect of cross-rolling during sheet properties on the mechanical properties of magnesium-zinc-cerium alloys.

## Magnesium-Zirconium Alloys

### Melting and Casting Alloys

Initially, two methods of adding zirconium were used. An alloying procedure recommended by Dow Chemical Company using TAM zirconium tetrachloride flux gave poor results as spectrographic analyses showed only a trace of zirconium present. Two melts were made using a master alloy containing 60 magnesium-40 zirconium. These alloys also exhibited poor recovery of zirconium. A comparison of analyses and mechanical properties gave no indication that one method was better than the other.

Two additional methods of adding zirconium were tried and the results were improved considerably. One method was to introduce zirconium as zirconium sponge and the second method used "dense" zirconium tetrachloride. Details of these methods are given in Appendix VI.

A summary of intended compositions and spectrographic analyses for magnesium-zirconium alloys is given in Table XVII. As noted, half of these alloys exhibited non-metallic inclusions and spectrographic analyses were not considered reliable. These inclusion- containing heats were not used for the determination of mechanical properties. The procedure for introducing the zirconium by means of dense zirconium tetrachloride was found to be less complicated and more adapted to the scale of melting operations in this development work. It has, therefore, been adopted as the standard practice for introducing zirconium in magnesium and magnesium-zinc alloys.

## TABLE XVII

### INTENDED COMPOSITIONS AND SPECTROGRAPHIC ANALYSES OF MAGNESIUM-ZIRCONIUM ALLOYS

| Alloy | Intended Zirconium Content | Spectrographic Analyses | Source of Zirconium |
|---|---|---|---|
| R-751 | 0.1 | 0.10 | dense zirconium tetrachloride |
| 752 | 0.1 | 0.09 | " " " |
| 753 | 0.1 | 0.20 | " " " |
| 768(a) | 0.1 | 0.1-0.3 | zirconium sponge |
| 762(a) | 0.5 | 0.15 | dense zirconium tetrachloride |
| 764(a) | 0.5 | 0.15-0.30 | " " " |
| 766 | 0.5 | 0.44 | " " " |
| 763(a) | 1.0 | 0.15-0.20 | dense zirconium tetrachloride |
| 765(a) | 1.0 | 0.40-0.45 | " " " |
| 767 | 1.0 | 0.73 | " " " |
| 769 | 1.0 | 0.76 | " " " |
| 770(a) | 1.0 | 0.54 | zirconium sponge |
| 771 | 1.0 | 0.59 | " " |
| 772 | 1.0 | 0.55 | " " |
| 773(a) | 1.0 | 0.60 | " " |

(a) Many flux inclusions were visible to the eye. These ingots were not used for determining mechanical properties.

Mechanical Properties

A study of the recrystallization data for hot and warm rolled sheet in this system indicated that the alloys did not respond to the fixed rolling conditions in the expected manner. Only alloy R-766 (magnesium-0.44 zirconium) exhibited a typical recrystallization curve. The as-rolled hardness values of alloys above and below 0.44% zirconium suggested that the structures were recrystallized during warm and hot rolling. A metallographic examination produced evidence in substantial agreement with this observation although it was considered that the extent of the examination was of limited scope. Accordingly, the average mechanical properties of the alloys, given in Table XVIII, are not believed to be representative of the capabilities of the system.

Plans for Future Work in This System

Plans for future work in this system are:

1. Investigate rolling conditions to establish suitable hot and warm rolling procedures.
2. Re-cast the alloys in this group for a new trial to produce hot and warm rolled sheet in stress relieved and low temperature annealed conditions.

## TABLE XVIII

## AVERAGE MECHANICAL PROPERTIES OF MAGNESIUM-ZIRCONIUM ALLOYS

| Alloy | Composition, Spectro. Anal. Zirconium | Nominal Rolling Temp. °F Hot | Warm | Heat Treatment Time | Temp. | Kips per sq. in. CYS | TYS | UTS | Elong. % 2 in. |
|---|---|---|---|---|---|---|---|---|---|
| 752Ws | 0.09 | 700 | 500 | 1 hr. | 300 | 15.0 | 13.6 | 23.7 | 4.5 |
| 752Wa(L) | - | 700 | 500 | 1 | 700 | 7.6 | 13.5 | 25.4 | 5.0 |
| 752Hs | - | 700 | - | 1 | 300 | 12.9 | 14.9 | 24.4 | 6.0 |
| 752Ha(L) | - | 700 | - | 1 | 600 | 10.7 | 15.0 | 25.7 | 5.5 |
| 751Ws | 0.10 | 700 | 500 | 1 | 300 | 12.2 | 13.1 | 24.5 | 6.0 |
| 751Hs | - | 700 | - | 1 | 300 | 12.2 | 13.2 | 24.9 | 7.5 |
| 751Ha(L) | - | 700 | - | 1 | 700 | 6.0 | 13.7 | 25.0 | 6.0 |
| 753Ws | 0.20 | 700 | 500 | 1 | 300 | 11.6 | 13.6 | 23.7 | 6.0 |
| 753Hs | - | 700 | - | 1 | 500 | 11.2 | 22.8 | 32.4 | 8.0 |
| 766Ws | 0.44 | 700 | 500 | 1 | 600 | 8.5 | 17.4 | 30.8 | 8.0 |
| 766Wa(L) | - | 700 | 500 | 1 | 700 | 7.3 | 16.8 | 30.6 | 11.0 |
| 766Hs | - | 700 | - | 1 | 475 | 22.3 | 22.8 | 32.4 | 7.5 |
| 766Ha(L) | - | 700 | - | 1 | 565 | 14.8 | 22.1 | 32.3 | 7.5 |
| 772Ws | 0.55 | 700 | 500 | 1 | 550 | 12.1 | 15.4 | 26.3 | 7.5 |
| 772Wa(L) | - | 700 | 500 | 1 | 750 | 6.8 | 13.3 | 25.4 | 6.0 |
| 772Hs | - | 700 | - | 1 | 600 | 10.1 | 13.1 | 27.0 | 7.5 |
| 772Ha(L) | - | 700 | - | 1 | 700 | 7.5 | 13.7 | 26.3 | 7.0 |
| 771Ws | 0.59 | 700 | 500 | 1 | 550 | 12.7 | 14.6 | 26.8 | 8.0 |
| 771Hs | - | 700 | - | 1 | 450 | 14.9 | 13.3 | 24.4 | 8.0 |
| 771Ha(L) | - | 700 | - | 1 | 600 | 9.8 | 12.0 | 26.6 | 7.5 |
| 767Ws | 0.73 | 700 | 500 | 1 | 500 | 13.4 | 13.8 | 25.8 | 8.5 |
| 767Wa(L) | - | 700 | 500 | 1 | 700 | 7.1 | 12.9 | 26.2 | 8.0 |
| 767Hs | - | 700 | - | 1 | 600 | 10.2 | 13.7 | 26.3 | 7.0 |
| 767Ha(L) | - | 700 | - | 1 | 750 | 6.9 | 13.4 | 27.3 | 7.5 |
| 769Ws | 0.76 | 700 | 500 | 1 | 700 | 7.9 | 13.5 | 26.0 | 7.5 |
| 769Hs | - | 700 | - | 1 | 500 | 12.7 | 14.5 | 25.6 | 9.0 |
| 769Wa(L) | - | 700 | - | 1 | 600 | 9.4 | 14.0 | 26.7 | 7.5 |

## Magnesium-Zinc-Zirconium Alloys

### Melting and Casting Alloys

The general procedure of melting and casting discussed for magnesium-zirconium alloys was applied to the limited amount of work carried out in this system. Although five alloys were prepared, a general condition of flux inclusions was present and spectrographic analyses for zirconium were considered unreliable. Additional experimental work was not conducted in order to devote more time to the magnesium-zinc-cerium alloy study.

### Plans for Future Work in this System

It is planned to prepare the following alloys for an initial survey of the mechanical properties in this system:

| Zinc | Zirconium |
|---|---|
| 0.8 | 0.2 |
| 0.5 | 0.5 |
| 0.2 | 0.8 |

If the results warrant the effort, an extension of the study will be made.

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# APPENDIX I

## DETAILED EXPERIMENTAL PROCEDURE FOR MELTING AND CASTING MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

The following sequence of operations was adopted as a standard procedure for melting and casting magnesium-lithium base alloys.

1. The alloy components were placed in the crucible and the apparatus was assembled. In this charging, it was believed to be more desirable to load the components with the lithium on top. This prevented "bridging" of the components above the melt and reduced the time during which the lithium was molten and not alloyed.
2. Argon was passed through the unit for 10 minutes to sweep out the air. During this period the packing gland was loosened and a steady flow of argon maintained. At the end of 10 minutes the packing gland was tightened and the argon pressure adjusted to 2 psi. This pressure was maintained for the remainder of the cycle.
3. The charge was heated until it became molten. The presence of solid components could be detected easily by moving the stirring rod up and down. When the charge was completely molten, the temperature was recorded to use as a guide in the remainder of the cycle.
4. The charge was completely mixed by stirring for 1 minute by slowly sliding the stirring rod up and down.

5. The charge was superheated to a minimum temperature of 1400°F. This was done to insure the complete alloying of the components and to increase the fluidity of the melt.

6. The charge was stirred for 1 minute to eliminate any compositional differences in the melt.

7. The temperature of the charge was dropped in the furnace to the pouring range. This operation took at least 15 minutes and allowed time for the non-metallics to settle.

8. The alloy was cast at approximately 50°F above the melting temperature. To cast the alloy the entire assembly was lifted from the furnace and slowly tilted so that the metal would run from the crucible to the mold. At the time of pouring the mold temperature had risen to the range 200-250°F by conduction from the crucible and housing.

9. The argon pressure of 2 psi was maintained for 5 minutes after casting to allow ample time for solidification. At the end of this period the apparatus was disassembled and the ingot was removed from the mold.

10. The crucible and stirring rod were cleaned by immersing them in dilute hydrochloric acid until the alloy which remained on them was dissolved. They were then rinsed in water and dried. The molds and housing were cleaned at less frequent intervals.

APPENDIX II

TABLE XIX

SUMMARY OF INTENDED COMPOSITIONS, CHEMICAL ANALYSES AND EXTRUSION CONDITIONS FOR EXPERIMENTAL MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-14 | 4.67 | 17.6 | - | | | | |
| L-15 | 8.9 | 10.0 | - | | | | |
| L-16 | 5.7 | 15.0 | - | | | | |
| L-17 | 7.85 | 11.0 | - | | | | |
| L-18 | 33.7 | 2.0 | - | 1.05 | - | 85.0 | 700-750 |
| L-19 | 19.0 | 5.0 | - | 4.87<br>4.90(i) | - | 70.0 | 700-750 |
| L-20 | 11.5 | 8.0 | - | 7.77 | - | 91.0 | 450-500 |
| L-21 | 7.3 | 12.0 | - | 12.25<br>11.85(j) | - | 14.0 | 700-750 |
| L-22 | 8.0 | 10.6 | 5.0 Al | 10.25<br>10.60(k) | 4.65 Al | 32.5 | 700-750 |
| L-23 | 8.0 | 10.0 | 10.0 Al | - | - | 54.5 | 700-750 |
| L-24 | 8.0 | 9.4 | 15.0 Al | - | - | 64.5 | 700-750 |
| L-25 | 8.0 | 8.9 | 20.0 Al | 8.54 | 18.9 Al | 80.0 | 700-750 |
| L-26 | 10.0 | 8.5 | 5.0 Al | 8.44 | 4.65 Al | 45.0 | 700-750 |
| L-27 | 10.0 | 8.2 | 10.0 Al | - | - | 74.5 | 700-750 |
| L-28(a) | 11.5 | 8.0 | - | - | - | - | - |

(▲) See notes at end of table.

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-29(a) | 7.3 | 12.0 | - | - | - | - | - |
| L-30(b) | 7.3 | 12.0 | - | - | - | - | - |
| L-31 | 10.0 | 7.8 | 15.0 Al | - | - | 89.5 | 700-750 |
| L-32(c) | 10.0 | 7.3 | 20.0 Al | - | - | - | - |
| L-33 | 10.0 | 8.9 | 2.0 Zn | - | - | 67.0 | 500-550 |
| L-34 | 10.0 | 8.7 | 4.0 Zn | - | - | 75.0 | 500-550 |
| L-35 | 10.0 | 8.5 | 6.0 Zn | - | - | 81.0 | 500-550 |
| L-36 | 10.0 | 8.4 | 8.0 Zn | 8.2 | 7.96 Zn | 86.0 | 500-550 |
| L-37 | 8.0 | 10.9 | 2.0 Zn | 10.75<br>11.3(k)<br>10.6(i) | 2.03 Zn | 42.0 | 500-550 |
| L-38 | 8.0 | 10.7 | 4.0 Zn | 10.65 | - | 46.5 | 500-550 |
| L-39 | 8.0 | 10.4 | 6.0 Zn | 10.75 | - | 51.5 | 500-550 |
| L-40 | 8.0 | 10.2 | 8.0 Zn | - | - | 61.0 | 500-550 |
| L-41 | 15.0 | 6.25 | - | 2.61 | - | 65.0 | 700-750 |
| L-42 | 15.0 | 6.1 | 2.0 Zn | 5.32 | 2.04 Zn | 75.6 | 700-750 |
| L-43 | 5.68 | 15.0 | - | 15.15<br>13.3(k) | - | 47.5 | 450-500 |
| L-44 | 4.0 | 20.0 | - | 19.25<br>15.3(k)<br>17.9(i) | - | 47.0 | 450-500 |
| L-45 | 15.0 | 6.0 | 4.0 Zn | - | - | 80.0 | 700-750 |
| L-46 | 15.0 | 5.9 | 6.0 Zn | - | - | 78.0 | 700-750 |
| L-47 | 17.0 | 5.9 | - | - | - | 78.0 | 700-750 |
| L-48 | 17.0 | 5.8 | 2.0 Zn | - | - | 89.0 | 700-750 |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-49 | 17.0 | 5.6 | 4.0 Zn | - | - | 87.0 | 700-750 |
| L-50 | 17.0 | 5.5 | 6.0 Zn | - | - | 88.5 | 700-750 |
| L-51 | 14.1 | 6.5 | 2.2 Al | 7.64 | 2.02 Al | 88.0 | 700-750 |
| L-52 | 17.0 | 5.6 | 4.0 Al | - | - | 96.0 | 750-800 |
| L-53 | 17.0 | 5.5 | 6.0 Al | - | - | 92.0 | 800-850 |
| L-54 | 17.0 | 5.8 | 2.0 Al | - | - | 90.0 | 750-800 |
| L-55 | 15.0 | 6.1 | 2.0 Al | 5.1 | 1.87 Al | 86.0 | 750-800 |
| L-56 | 15.0 | 6.0 | 4.0 Al | 4.70 | 3.87 Al | 95.0 | 750-800 |
| L-57 | 15.0 | 5.9 | 6.0 Al | 3.56 | 6.14 Al | 93.0 | 800-850 |
| L-58 | 6.0 | 13.6 | 5.0 Al | 12.6<br>12.8(k)<br>13.1(i) | 5.06 Al | 55.0 | 700-750 |
| L-59 | 6.0 | 12.9 | 10.0 Al | - | - | 54.0 | 700-750 |
| L-60 | 6.0 | 12.2 | 15.0 Al | - | - | 54.5 | 700-750 |
| L-61 | 6.0 | 11.4 | 20.0 Al | - | - | 69.4 | 700-750 |
| L-62(d) | - | - | 45.4 Al | - | - | - | - |
| L-63(e) | - | 20.47 | 79.53 Al | - | - | - | - |
| L-64 | 6.0 | 13.6 | 5.0 Cd | - | - | 37.5 | 500-550 |
| L-65 | 6.0 | 12.9 | 10.0 Cd | - | - | 46.5 | 500-550 |
| L-66 | 6.0 | 12.2 | 15.0 Cd | - | - | 53.0 | 500-550 |
| L-67 | 6.0 | 11.4 | 20.0 Cd | - | - | 61.0 | 500-550 |
| L-68 | 30.0 | 3.2 | - | - | - | 54.5 | 750-800 |
| L-69 | 30.0 | 3.2 | 2.0 Al | 1.93 | 1.65 Al | 81.0 | 750-800 |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-70 | 30.0 | 3.1 | 4.0 Al | 2.75 | 3.65 Al | 75.0(f) | 750-800 |
| L-71 | 30.0 | 3.0 | 6.0 Al | - | - | 89.0(f) | 750-800 |
| L-72 | 99.0 | 1.0 | 2.0 Al | - | - | 61.5(g) | 750-800 |
| L-73 | 99.0 | 1.0 | 4.0 Al | - | - | 67.1(g) | 750-800 |
| L-74 | 99.0 | 0.9 | 6.0 Al | - | - | 72.0(g) | 750-800 |
| L-75 | - | - | 2.0 Al | - | - | 59.2(g) | 750-800 |
| L-76 | - | - | 4.0 Al | - | - | 75.0(g) | 750-800 |
| L-77 | - | - | 6.0 Al | - | - | 77.1(g) | 750-800 |
| L-78 | - | - | 2.0 Zn | - | - | 58.0(g) | 750-800 |
| L-79 | - | - | 4.0 Zn | - | - | 61.6(g) | 750-800 |
| L-80 | - | - | 6.0 Zn | - | - | 58.7(g) | 750-800 |
| L-81 | 99.0 | 1.0 | 2.0 Zn | - | - | 63.5(g) | 750-800 |
| L-82 | 99.0 | 1.0 | 4.0 Zn | - | - | 60.2(g) | 750-800 |
| L-83 | 99.0 | 0.9 | 6.0 Zn | - | - | 60.0(g) | 750-800 |
| L-84 | 30.0 | 3.2 | 2.0 Zn | - | - | 86.3 | 750-800 |
| L-85 | 30.0 | 3.1 | 4.0 Zn | - | - | 83.6 | 750-800 |
| L-86(c) | 30.0 | 3.0 | 6.0 Zn | - | - | - | - |
| L-87 | 6.0 | 14.0 | 2.2 Zn | - | - | 34.0 | 700-750 |
| L-88 | 6.0 | 12.9 | 10.0 Zn | - | - | 40.0 | 700-750 |
| L-89 | 6.0 | 12.2 | 15.0 Zn | - | - | 42.5 | 700-750 |
| L-90 | 6.0 | 11.4 | 20.0 Zn | - | - | 35.0 | 700-750 |
| L-91 | 8.12 | 11.0 | - | - | - | 50.0 | 450-500 |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-92 | 9.0 | 10.0 | - | - | - | 54.0 | 450-500 |
| L-93(l) | - | 7.2 | 67.5 Zn | - | - | - | - |
| L-94(m) | - | 13.4 | 63.2 Zn | - | - | - | - |
| L-95(n) | - | - | 72.9 Zn | - | - | - | - |
| L-96 | 7.0 | 12.3 | 2.0 Zn | - | 1.8 Zn | 70.0 | 450-500 |
| L-97 | 7.0 | 12.0 | 4.0 Zn | - | - | 90.0 | 450-500 |
| L-98 | 7.0 | 11.7 | 6.0 Zn | - | - | 130.0 | 450-500 |
| L-99 | 7.0 | 11.5 | 8.0 Zn | - | - | 120.0 | 450-500 |
| L-100 | 7.0 | 11.2 | 10.0 Zn | - | - | 100.0 | 450-500 |
| L-101(o) | 7.0 | 10.6 | 15.0 Zn | - | - | - | - |
| L-102 | 7.0 | 10.0 | 20.0 Zn | - | - | 190.0 | 450-500 |
| L-103(o) | 8.0 | 10.0 | 10.0 Zn | - | - | - | - |
| L-104(o) | 8.0 | 9.8 | 12.0 Zn | - | - | - | - |
| L-105(o) | 8.0 | 9.5 | 15.0 Zn | - | - | - | - |
| L-106(o) | 8.0 | 8.9 | 20.0 Zn | - | - | - | - |
| L-107(o) | 10.0 | 8.2 | 10.0 Zn | - | - | - | - |
| L-108(o) | 10.0 | 8.0 | 12.0 Zn | - | - | - | - |
| L-109 | 10.0 | 7.8 | 15.0 Zn | 16.1 | - | 136.0 | 450-500 |
| L-110(o) | 10.0 | 7.3 | 20.0 Zn | - | - | - | - |
| L-111 | 15.0 | 5.8 | 8.0 Zn | - | - | 148.0 | 550-600 |
| L-112 | 15.0 | 5.6 | 10.0 Zn | - | - | 144.0 | 600-650 |
| L-113 | 15.0 | 5.3 | 15.0 Zn | - | - | 140.0 | 600-650 |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-114 | 15.0 | 5.0 | 20.0 Zn | - | - | 150.0 | 600-650 |
| L-115(p) | 30.0 | 3.0 | 8.0 Zn | - | - | - | - |
| L-116 | 30.0 | 2.9 | 10.0 Zn | - | - | 170.0 | 550-600 |
| L-117 | 30.0 | 2.7 | 15.0 Zn | - | - | 160.0 | 600-650 |
| L-118 | 30.0 | 2.6 | 20.0 Zn | - | - | 180.0 | 600-650 |
| L-119 | 30.0 | 3.0 | 8.0 Zn | - | - | 150.0 | 600-650 |
| L-120 | 9.83 | 9.0 | 2.5 Al | - | - | 56.0 | 500-700 |
| L-121 | 8.28 | 10.5 | 2.5 Al | 9.85 | - | 80.0 | 500-600 |
| L-122 | 7.13 | 12.0 | 2.5 Al | 11.75 | - | 70.0 | 500-600 |
| L-123 | 10.3 | 8.5 | 4.0 Al | 7.95 | 5.9 Al | 120.0 | 600-650 |
| L-124 | 7.27 | 11.5 | 5.0 Al | 10.3 | 5.3 Al | 60.0 | 600-650 |
| L-125(q) | - | - | - | - | - | - | - |
| L-126 | 6.83 | 11.5 | 10.0 Al | - | - | 64.0 | 600-650 |
| L-127 | 6.73 | 11.0 | 15.0 Al | - | - | 100.0 | 600-650 |
| L-128 | 6.52 | 12.5 | 6.0 Al | - | - | 64.0 | 600-650 |
| L-129 | 6.36 | 12.5 | 8.0 Al | - | - | 60.0 | 600-650 |
| L-130 | 20.8 | 4.5 | 2.0 Al | - | - | 150.0 | 600-650 |
| L-131 | 31.3 | 3.0 | 3.0 Al | - | - | 160.0 | 600-650 |
| L-132 | 17.0 | 5.0 | 10.0 Al | - | - | 160.0 | 600-650 |
| L-133 | 14.0 | 6.0 | 10.0 Al | - | - | 180.0 | 600-650 |
| L-134 | 11.9 | 7.0 | 10.0 Al | - | - | 174.0 | 600-650 |
| L-135 | - | - | 10.0 Al | - | - | 140.0 | 600-700 |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-136 | 29.0 | 3.0 | 10.0 Al | - | - | 100.0 | 650-700 |
| L-137(r) | - | 34.0 | 66.0 Al | - | - | - | - |
| L-138 | 1.64 | 19.3 | 49.1 Al | - | - | 164.0 | 600-700 |
| L-139 | 100.0 | 8.5 | 6.0 Al | - | - | 100.0 | 650-700 |
| L-140 | - | - | 7.5 Al | - | - | 160.0 | 650-700 |
| L-141 | 91.0 | 1.0 | 8.0 Al | - | - | 160.0 | 500-700 |
| L-142 | 7.73 | 11.0 | 4.0 Al | - | - | 70.0 | 500-700 |
| L-143 | 16.0 | 5.0 | 15.0 Al | - | - | 150.0 | 500-700 |
| L-144 | 23.3 | 3.5 | 15.0 Al | - | - | 150.0 | 500-700 |
| L-145 | 15.0 | 5.0 | 20.0 Al | - | - | 150.0 | 500-700 |
| L-146 | 21.9 | 3.5 | 20.0 Al | - | - | 160.0 | 500-700 |
| L-147(s) | 5.33 | 15.0 | 5.0 Al | - | - | - | - |
| L-148 | 5.20 | 15.0 | 7.0 Al | - | - | 110.0 | 550-600 |
| L-149 | 5.07 | 15.0 | 9.0 Al | - | - | 88.0 | 550-600 |
| L-150 | 3.80 | 20.0 | 4.0 Al | - | - | 150.0 | 450-500 |
| L-151 | 3.70 | 20.0 | 6.0 Al | - | - | 104.0 | 550-600 |
| L-152 | 3.60 | 20.0 | 8.0 Al | - | - | 110.0 | 550-600 |
| L-153 | 4.67 | 15.0 | 15.0 Al | - | - | 70.0 | 500-700 |
| L-154 | 4.33 | 15.0 | 20.0 Al | - | - | 60.0 | 500-700 |
| L-155 | 3.25 | 20.0 | 15.0 Al | - | - | 70.0 | 500-700 |
| L-156(o) | 7.0 | 10.6 | 15.0 Zn | - | - | - | - |
| L-157(o) | 8.0 | 10.0 | 10.0 Zn | - | - | - | - |

| Alloy Number | Intended Composition (Weight) Mg/Li Ratio | % Li | Third Element | Analyses (Wt.) % Li | Third Element | Extrusion Conditions(h) Force (kips) | Temp. °F |
|---|---|---|---|---|---|---|---|
| L-158 | 8.0 | 9.8 | 12.0 Zn | - | 11.7 Zn<br>11.7 Zn | 60.0 | 500-700 |
| L-159 | 8.0 | 9.5 | 15.0 Zn | - | - | 60.0 | 500-700 |
| L-160 | 8.0 | 8.9 | 20.0 Zn | - | - | 60.0 | 500-700 |
| L-161 | 10.0 | 8.2 | 10.0 Zn | - | - | 60.0 | 500-700 |
| L-162 | 10.0 | 8.0 | 12.0 Zn | - | - | 60.0 | 500-700 |
| L-163 | 10.0 | 7.3 | 20.0 Zn | - | - | 70.0 | 500-700 |
| L-164 | 30.0 | 3.0 | 8.0 | - | - | 150.0 | 500-700 |

(a) Used for metallographic study.
(b) Alloy made with the addition of 75 LiCl-25 LiF flux.
(c) Not extruded.
(d) Intended to be $Mg_{17}Al_{12}$ phase.
(e) Intended to be AlLi phase.
(f) Extruded to 0.170" diameter rod, extrusion ratio 75.
(g) Extruded to 0.250" diameter rod, extrusion ratio 36.
(h) Extrusion conditions for extrusion to 0.125" diameter rod, extrusion ratio 144 except as noted.
(i) Analysis since last final report, 7-27-50.
(j) Analysis since last final report, 1-3-50.
(k) Analysis since last final report, 3-7-50.
(l) Intended to be MgLiZn phase.
(m) Intended to be $MgLi_2Zn$ phase.
(n) Intended to be MgZn.
(o) Lost in homogenization.
(p) Poor casting - scrap.
(q) Not made.
(r) Intended to be $AlLi_2$.
(s) Scrap.

## APPENDIX III

## CALCULATION OF LATTICE PARAMETERS FOR HEXAGONAL CLOSE-PACKED STRUCTURES

### Assigning Indices to Low-Angle Transmission Lines

For normal HCP patterns (c/a = approx. 1.62), the order of appearance of lines on the film is as follows:

| | |
|---|---|
| (100) | (102) |
| (002) | (110) |
| (101) | (103) |

These lines can be readily assigned proper indices by inspection or by comparing measured and calculated $\sin^2\theta$ values for tentative assignment of indices.

Note: The structure factor = 0. Hence, there is no diffraction line if L = an odd integer and (h + 2 k) = 3 n, where n = 0, 1, 2, 3, ---- for a normal HCP plane of indices (h k · L).

### Calculation of Approximate $a_o$ Value

Use $\sin^2\theta$ values (from film measurements) for two lines of known (h k · L) as determined in the first section of this Appendix III above, for which L = 0 such as (100) and (110).

The basic equation is:

$$\sin^2\theta = \frac{\lambda^2}{4}\left[\frac{4}{3}\left(\frac{h^2 + hk + k^2}{a_o^2}\right) + \frac{L^2}{c_o^2}\right]$$

Then:

$$\sin^2\theta_{(110)} - \sin^2\theta_{(100)} =$$

$$\frac{\lambda^2}{3a_o^2}\left[(h^2 + hk + k^2)_{110} - (h^2 + k^2 + hk)_{100}\right]$$

Solve this for $a_o$

Calculation of Approximate $c_o$ Value

Use measured $\sin^2\theta$ values for two known lines of similar h and k values such as (101) and (102).

Then:

$$\sin^2\theta_{(102)} - \sin^2\theta_{(101)} = \frac{\lambda^2}{4c_o^2}\left[L^2_{(102)} - L^2_{(101)}\right]$$

Solve this for $c_o$

Note: For lines where the $K\alpha_1$, $K\alpha_2$ doublet is not resolved, use a wave length $\lambda$ obtained by weighting $\lambda\alpha_1$ twice as much as $\lambda\alpha_2$, since darker part of the line will be nearer the $\alpha_1$ position.

Thus, $\lambda = \frac{1}{3}\left[2\,\lambda\alpha_1 + \lambda\alpha_2\right]$

Assigning Indices to Back-Reflection Lines

The basic equations are:

$$\frac{1}{d^2} = \frac{4}{3}\left[\frac{h^2 + hk + k^2}{a_o^2}\right] + \left[\frac{L}{c_o}\right]^2$$

$$d = \frac{\lambda}{2\sin\theta}$$

Then:

$$a_o = \frac{\lambda}{2\sin\theta}\left[\frac{4}{3}(h^2 + hk + k^2) + \frac{L^2}{(c_o/a_o)^2}\right]^{1/2}$$

To use this equation with known $\lambda$ (see note above) and measured $\sin\theta$ values:

a. Insert approximate values for $a_o$ and $c_o$ from II and III.

b. Since h, k, L and $(h^2 + hk + k^2)$ must all be integral values, assume L = 0, 1, 2, 3, ---- etc. successively and from the above equation for each value of L, calculate the

value of $(h^2 + hk + k^2)$.

c. At the correct value of L for the line in question, $(h^2 + hk + k^2)$ will be an integral value (or very close to it) and the proper h and k values can be assigned for the line by inspection.

Example of Method:

Unknown line has $\theta = 50°$ (measured from film), approximate $a_o = 3.20$, $c_o/a_o = 1.62$ (calculated from transmission lines), $\lambda = 1.5387$.

Then: $$3.20 = \frac{1.5387}{2 \sin 50°} \left[\frac{4}{3}(h^2 + hk + k^2) + \frac{L^2}{(1.62)^2}\right]^{1/2}$$

For L = 0, $(h^2 + hk + k^2) = 7.7$

L = 1, " $= 7.4$

L = 2, " $= 6.5$

L = 3, " $= 5.1$ } Nearest to integral value

L = 4, " $= 3.1$ }

By inspection, L cannot = 3 because no integral values of h and k can make $(h^2 + hk + k^2) = 5$. Therefore L must = 4 and $h = 1$, $k = 1$; $(h^2 + hk + k^2) = 3$.

Thus the unknown line at $\theta = 50°$ is (11.4).

Determination of Accurate $a_o$ and $c_o$ Values from Back-Reflection Lines

Use any two lines for which indices have been determined as above but preferably of highest possible $\theta$ values for which measured $\sin^2\theta$ values can be obtained.

The basic equation is:

$$\sin^2\theta = \frac{\lambda^2}{4}\left[\frac{4}{3}\left(\frac{h^2 + hk + k^2}{a_o^2}\right) + \frac{L^2}{c_o^2}\right]$$

For any two lines of known indices insert known values for:

$\sin^2\theta$

$\lambda$

$(h^2 + hk + k^2)$

$L^2$

and solve the two resulting equations simultaneously for $a_o$ and $c_o$.

# APPENDIX IV

## METALLOGRAPHIC TECHNIQUE FOR MAGNESIUM-LITHIUM BASE TERNARY ALLOYS

### Specimen Mounting

The successful use of microscopic examination of quenched specimens as a method for establishing phase boundaries depends on the retention, at room temperature, of the equilibrium microstructure obtained at some elevated temperature of interest. Since the 1/8" diameter wire specimens required mounting for handling, it was necessary to consider specimen mounting procedures which would minimize the alteration of the quenched condition by reheating during this preparation for polishing.

The use of lucite and bakelite, curing under pressure and in a temperature range approximately 130 to 175°C (266-345°C), were considered as second choice methods. Solders, waxes, mixtures of litharge and glycerin and clamp type mountings all have limitations in usefulness as a medium for specimen mounting.

A solution to the problem was found in the use of "Selectron 5003"(a), a thermosetting resin capable of being "cast" to hold any shape of specimen without pressure, curing at room temperature. Mounts of cured "Selectron" develop a hardness similar to lucite, respond to rough preparation similar to bakelite, have good melting characteristics for edge preservation, develop a high polish, are resistant to most commonly used etch-

(a) Commercial Trade Name, produced by Pittsburgh Plate Glass Co., Pittsburgh, Pa.

ants and do not require elaborate equipment for processing. The most significant disadvantage - the much longer time required to cure the mount than one of the phenolic resin type - can be offset to some degree by the simultaneous preparation of any number of mounts at one time. In practice, when making a large number of mounts, the method is considerably faster than lucite or bakelite mounting.

The principal steps in the mounting operation are outlined below.

1. A suitable mold for casting "Selectron" around specimen sections was found to be NO 101 Dixie cups, a small waxed paper cup approximately 1" diameter at the base and 1 1/2" high. The polymerized mount is removed easily from this expendable and inexpensive cup.
2. Selectron 5003, a syrupy liquid, is mixed in the proportion 100 ml. Selectron to 5 ml. of a catalyst, tertiary butyl hydroperoxide, just prior to use.
3. The mixture is "cast" into the mold with the transverse and longitudinal sections to be examined in position in the cup.
4. The casting resin will polymerize at room temperature in a period of two to three days leaving a tacky surface layer which may be removed easily. Although not desired for the phase boundary study, polymerization may be accelerated by heating to a low temperature. For example, a mount may be cured in approximately 4 hours

at 80°C (185°F).

A photograph of the resin, catalyst, mold and completed specimen is shown in Fig. 34.

Rough Preparation

The mounted specimens are drawn in one direction across a sharp file similar to the type used for filing soft metals (for example, an automobile body bumping file). The extremely sharp teeth and the wide spacing simulate a microtome effect and remove the effects of distortion resulting from the sawing operation. The file is cleaned frequently with a file card during the operation.

When a flat surface has been obtained the specimens are ground successively on #1, #1/0, #2/0 and #3/0 dry metallographic emery papers using a few drops of paraffin dissolved in kerosene as a lubricant to prevent smudging and surface distortion. Specimens are drawn in one direction across the papers placed over a plate glass for support and the operation is continued only long enough to remove the evidence of the preceding operation.

Specimens are then washed in soap and water to remove the lubricant and abrasive particles.

Rough Polishing

Rough polishing is accomplished on a "Vel-Chamee"(a) cloth charged with a water suspension of #600 alundum flour, to which is added 50 ml. of liquid soap per liter of suspension. The abrasive is flooded on the wheel during the operation which

(a) Supplied by John Ritzenthaler, 73 Franklin Square, New York 13, N.Y.

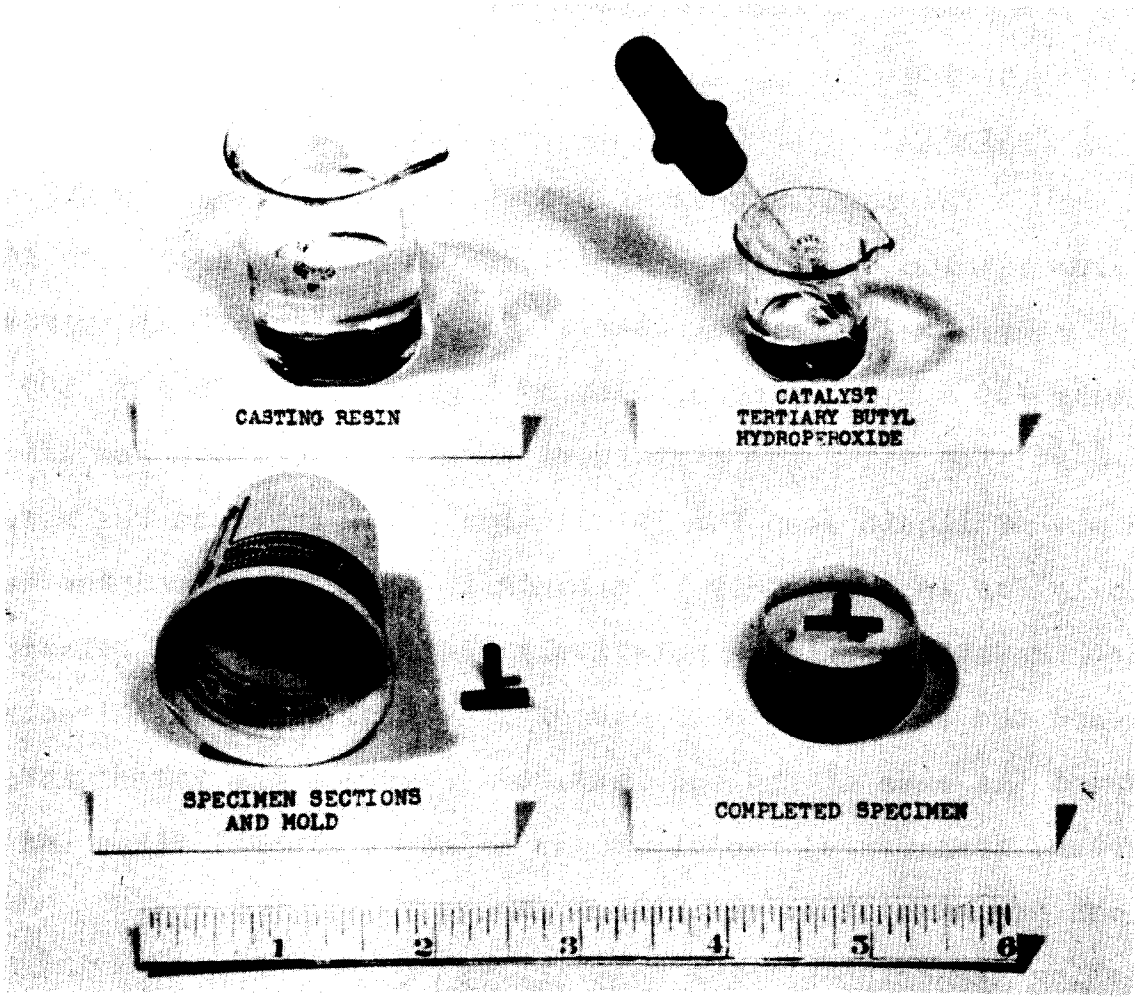



Figure 34

Materials required for preparing a specimen for microscopic examination and polystyrene resin at room temperature and without pressure.

requires approximately 10 seconds at a speed of 1200 RPM. Specimens are rinsed in absolute alcohol, blown dry and examined under the microscope.

Final Polishing

The most successful procedure for final polishing magnesium-lithium base alloys employed a suspension of "Linde A" - 5175 alumina(a) on a "Vel Chamee" No. 5 wheel cloth at approximately 400 R.P.M. A suspension of 4 grams of Linde A in 500 ml. of distilled water was a standard abrasive, to which was added 25 ml. of liquid soap for lubrication. The specimen was rotated slowly for approximately 10 seconds in a direction counter to the wheel rotation, using a relatively heavy pressure and relaxing toward the end. A thin stream of distilled water was directed toward the base of the specimen, the specimen was lifted into the stream, transferred rapidly to an adjacent alcohol rinse and blown dry with clean compressed air.

Etching Magnesium-Lithium Base Alloys

The general procedure for etching was to employ an immersion technique for a controlled length of time. All solutions used up to this time were at room temperature and specimens were agitated continually while immersed. After etching, specimens were transferred immediately to a small stream of absolute ethyl alcohol and blown dry with filtered compressed air.

The singly most important type of etchant for these alloys was "acetic picral" of the following "standard" formula:

(a) Supplied by Linde Air Products.

100 parts of 6% (by weight) piric acid in absolute ethyl alcohol

5 parts of glacial acetic acid

10 parts of distilled water.

This solution was applied to all alloys in an examination of structure and it was found to be generally applicable to magnesium-lithium-aluminum and magnesium-lithium-zinc alloys to reveal grain boundaries and distinguished between alpha and beta phases. The etching characteristics of standard acetic picral for the phases present in these two systems were as follows:

1. alpha phase- appeared clear and white, unstained, usually twinned and with fair to good definition of grain boundaries.
2. beta phase - appeared in various shades of brown to blue-black, usually attacked and stained. Grain boundaries were visible in most cases.
3. intermediate phases - in either of the two systems the intermediate phases were clear, ivory white, unattacked, usually heavily outlined from the matrix and unattacked.

One serious limitation of this etchant was the inability to produce a color distinction between the alpha phase and the intermediate phases. A considerable amount of time was devoted to a study of various modifications of the standard acetic picral as well as to the development of additional types of etchants to distinguish between alpha and intermediate phases. The results of these developments are described separately for each system.

Etching Magnesium-Lithium-Aluminum Alloys

A modification of the standard picral-acetic solution was developed which produced a clear distinction between the alpha and intermediate phases in certain ranges of alloy composition. The modification was the addition of 10 ml. of a 10% aqueous solution of tartaric acid to the standard picral-acetic acid solution. These results have been observed:

1. alpha phase - appeared various shades of grey to pink.
2. beta phase - appeared dark blue-brown to black.
3. intermediate phase - appeared clear to white.

This color distinction between the two clear phases was of considerable value in promoting the ease and accuracy of estimating the extent of the intermediate phase present. A comparison of the etching effect of standard acetic picral and tartaric acid modified acetic picral is shown in Figs. 35 and 36.

The tartaric acid modified solution was found to be the most useful in cases where alpha, beta and AlLi phases were present simultaneously. When the beta phase was absent, etching times became excessively long. As a general observation, it is believed that the main use of the tartaric acid addition will be to supplement the observations of the standard picral acetic acid reagent when AlLi phase is present.

An etchant composed of:

10 parts of 48% HF

90 parts of distilled water

was used to provide contrast between the phases alpha and

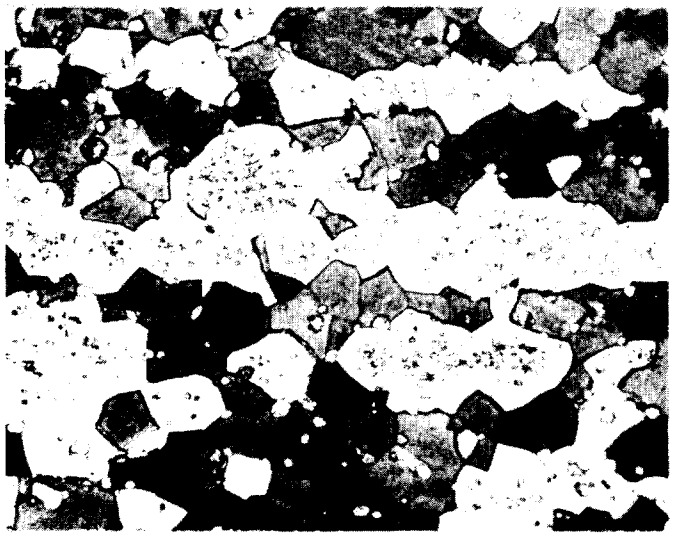


Figure 35

Neg: 249
Spec: M694

Mag: 500X
Etch: 100:5:10 Picral: Glacial Acetic Acid: Water, 8 sec.

Longitudinal section of alloy L-26 (magnesium-8.44 lithium-4.65 aluminum) heat treated 72 hours at 500°F and quenched in kerosene. The beta phase appears dark. The alpha and intermediate phases appear light, not clearly separated.

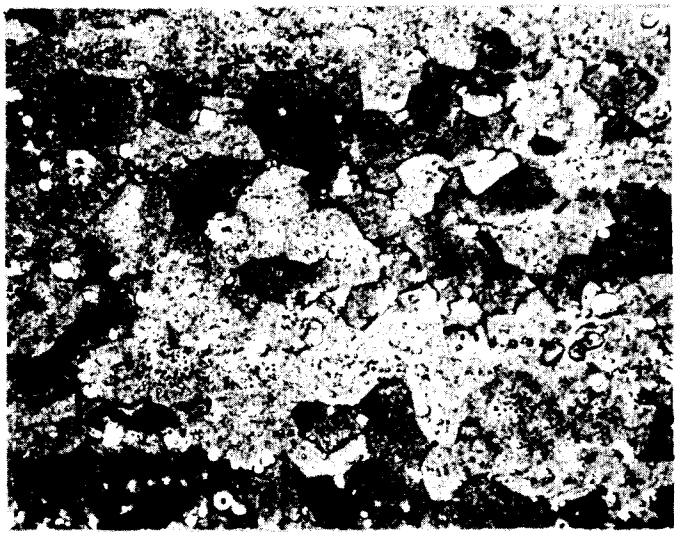


Figure 36

Neg: 248
Spec: M694

Mag: 500X
Etch: 100:5:10 Picral: Glacial Acetic Acid: Water plus 10 ml. of 10% tartaric acid.

Same structure as shown in Figure 35. The modified solution produces a color distinction between alpha and the intermediate phase.

$Mg_{17}Al_{12}$. With this etchant the phase $Mg_{17}Al_{12}$ was colored light brown and the alpha phase remained white. A dark product of the etching reaction tended to form in excessive amounts at the alpha grain boundaries. When the etchant was applied to alloys containing the AlLi phase, the AlLi particles were not clearly resolved.

In order to produce a distinction between the phases AlLi and $Mg_{17}Al_{12}$ when occurring in an alpha matrix, an etchant was developed containing the following:

100 parts of 10% tartaric acid in water

5 parts of glacial acetic acid.

This etchant colored the alpha phase gray while the $Mg_{17}Al_{12}$ and AlLi particles remained clear and white. The distinction between AlLi and $Mg_{17}Al_{12}$ phases was on the basis of surface attack at the boundaries between these phases and the alpha matrix. The boundary between the alpha and $Mg_{17}Al_{12}$ is sharp and clear while the boundary between the AlLi particles and the alpha is rough and irregular, giving a mottled appearance. One limitation of this etchant is that a product of the etching reaction tends to form in excessive amounts at the alpha grain boundaries.

Standard acetic picral was used with some success on alloys containing the phases alpha, AlLi, and $Mg_{17}Al_{12}$. The alpha phase was colored pink and orange while the intermediate phases remained clear and white. The AlLi particles were attacked heavily at their boundaries. The $Mg_{17}Al_{12}$ areas were evenly and distinctly outlined.

At the present time no single etchant has been found which will distinguish clearly the phases alpha, $Mg_{17}Al_{12}$ and AlLi when present simultaneously. This distinction becomes increasingly difficult when the relative amounts of the two intermediate phases are small. In this situation the procedure was to use several etchants (acetic picral, tartaric-acetic and HF in water) and to estimate the kind and mount of phases present under each condition of etching. This method was considered to give a reasonably accurate estimate of the phase distribution.

Etching Magnesium-Lithium-Zinc Alloys

In general, the modified standard acetic-picral solutions used in the magnesium-lithium-aluminum system were not applicable in this system.

The use of standard acetic-picral for alloys having a predominantly beta structure was not suitable due to severe staining of the beta phase. For these alloys the following etchant was useful:

10 gm. salicylic acid

100 ml. ethyl alcohol.

The etching characteristics of the salicylic acid solution were:

1. alpha phase - clear, white, unattacked without grain boundary definition or revelation of twinning planes.
2. beta phase - light brown without staining; good grain boundary definition.
3. MgLiZn phase - clear, white, unattacked.

The following etching solution was developed to detect small quantities of MgLiZn, even in the presence of the alpha phase.

| | |
|---|---|
| 100 ml. | $H_2O$ (distilled) |
| 1 gm. | $KMnO_4$ |
| 10 drops | $H_2SO_4$ (concentrated). |

The characteristics of this etchant were as follows:

1. alpha phase - shades of grayish-blue; stained.
2. beta phase - blue-black to black, heavily attacked and stained.
3. MgLiZn phase - white, unattacked, heavily outlined.

The permanganate solution, because of its vigorous action on the alpha and beta phases, was used only to supplement the information obtained with the standard acetic-picral and salicylic acid solutions. It was observed that potassium permanganate decomposes when added to an alcoholic solution so it could not be used as an addition to standard acetic-picral or the salicylic acid solutions.

## APPENDIX V

## DETAILS OF CONSTRUCTION OF MODIFICATION OF ELECTRICAL RESISTIVITY APPARATUS

### Specimen Container and Container Cover

The original container, container cover and container sealing system was replaced by a completely new cover and a revised sealing flange construction.

Considerable thought was given to the selection of an aluminum alloy for the cover and top section of the specimen container. Alcoa alloy #356 (Al-7 Si-3 Mg) was chosen as having the best weldability and retention of strength at elevated temperatures.

A section of the original container extending approximately 2 in. below the top was removed and a new section, machined from an annular cast ring of alloy #356, waswelded to the original 2S alloy container. The whole operation was planned so that no change in the original overall dimensions would result from the modifications. The new cylinder top section was made 1/16" greater in wall thickness than the original container so that more contact area would be available for gasketing between the cover and container. Details of the modified container are shown in Fig. 37.

Studs for securing the cover to the container were 1/4" - 20 X 2" stainless steel machine screws with the heads removed. Eight studs were placed around the circumference of

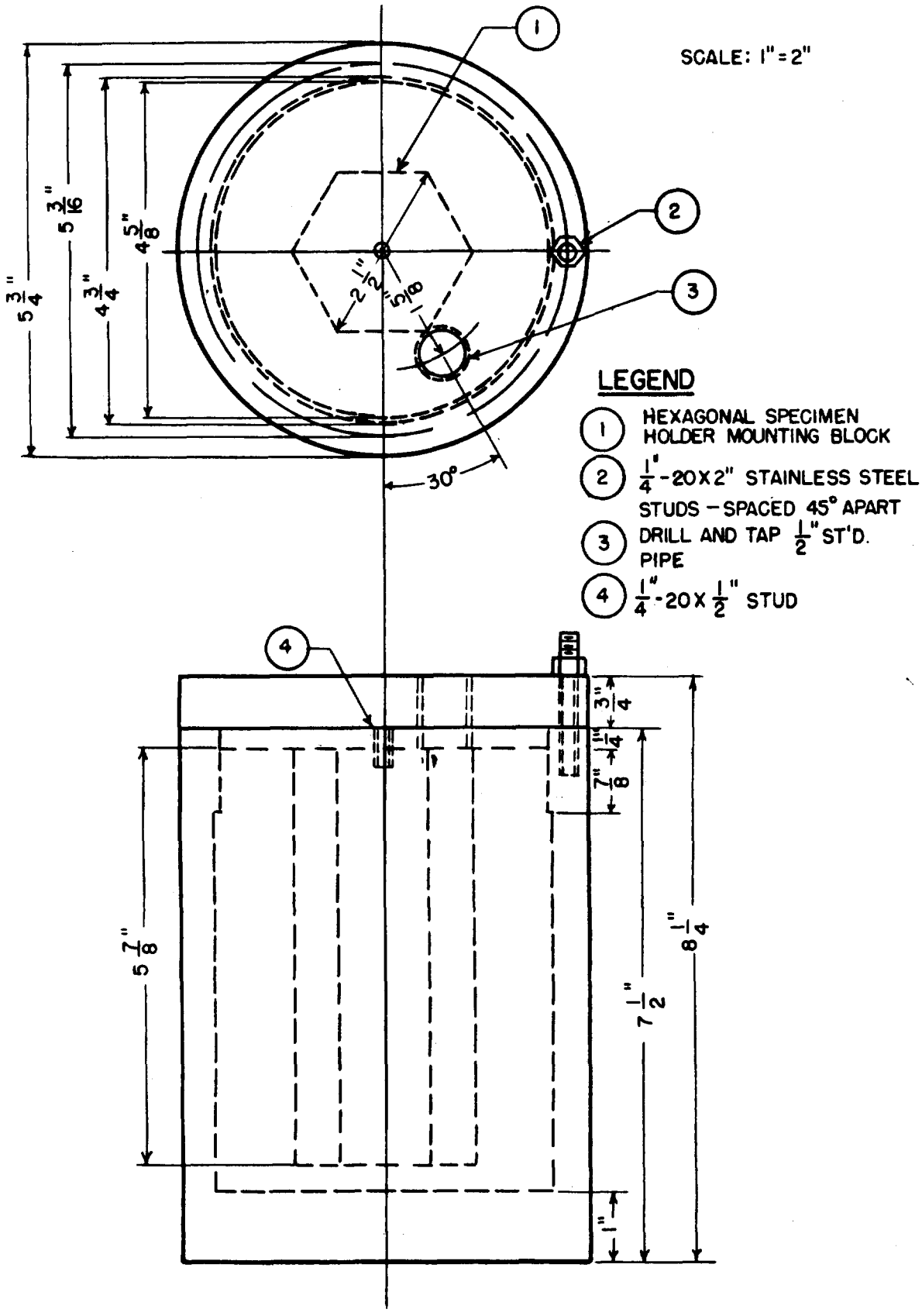



FIGURE 37

SEALED ALUMINUM SPECIMEN CONTAINER FOR ELECTRICAL RESISTIVITY APPARATUS

the cylinder at intervals of 45°. This arrangement was considered to give the optimum sealing pressure without weakening the container or depleting the gasket area excessively.

A new, solid cover for the specimen container was machined from a cast disk of alloy #356 to replace the original cover. In order to secure the best possible sealing conditions, the cover was recessed into the container and a fit of very close tolerance was maintained between the cover and specimen container wall. A single hole was made in the cover, drilled and topped for 1/2" steel pipe, to receive a nipple containing electrical circuit wires and gas inlet pipe. Refer to Fig. 37.

Electrical Terminal Head Assembly

The original system of electrical circuit wire leads from the container was replaced with a completely enclosed pipe pot-head system. The electrical connections were led, by the pipe system, to a point sufficiently distant from the container cover, so that temperatures were low enough to employ soldered wire terminals. An assembly drawing of the system is shown in Fig. 38 and a photograph of the completed construction is given in Fig. 12.

Referring to Fig. 38, a 1/2 in. x 4 1/2 in. standard pipe nipple was mounted vertically in the specimen container cover and was connected at the opposite end to a 1/2 in. standard pipe "tee". A 1/2 in. standard pipe plug with a centrally located 3/16 in. diameter hole was inserted into the vertical outlet of the "tee". A sturdy, gas-tight atmosphere inlet pipe was

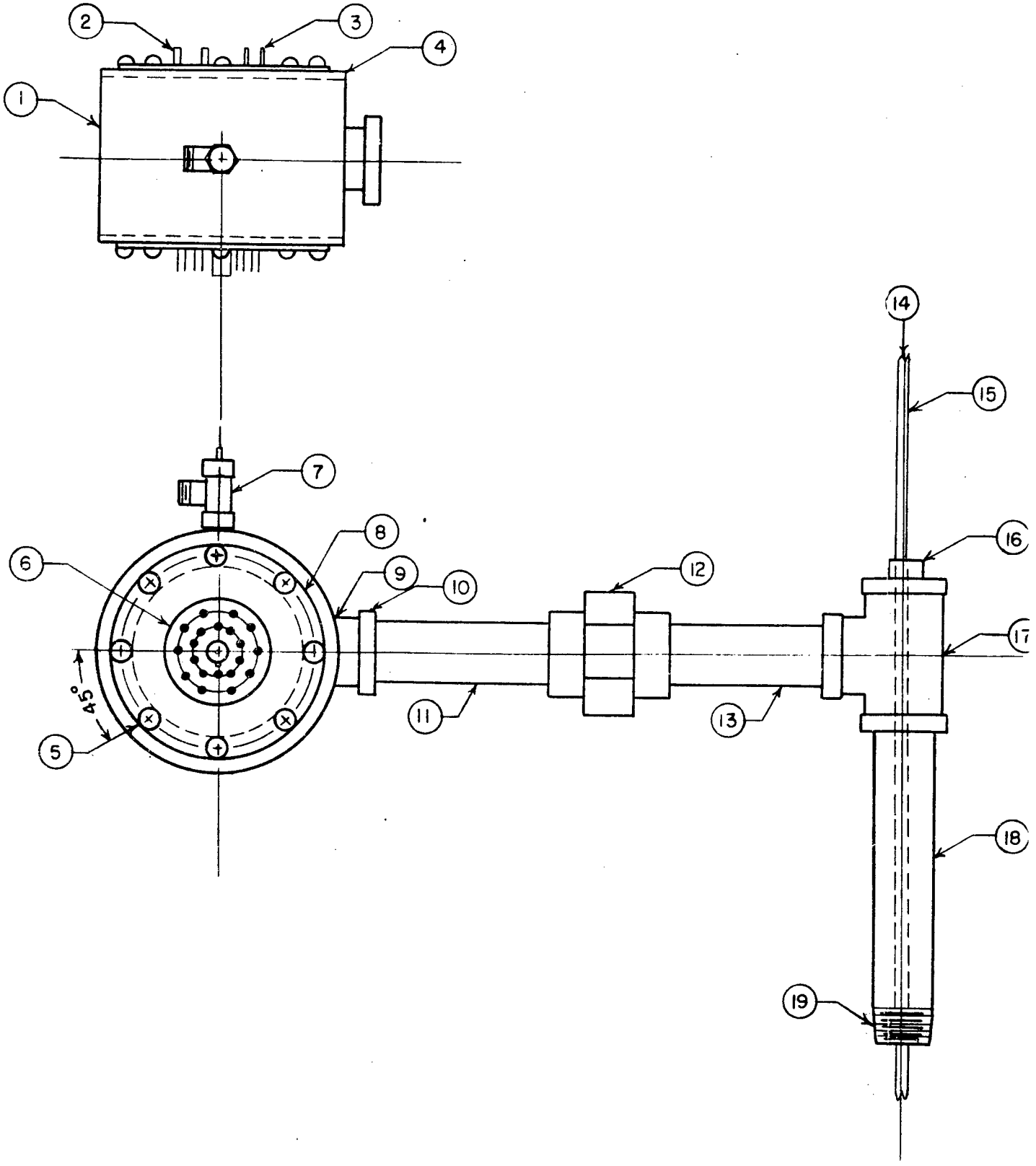


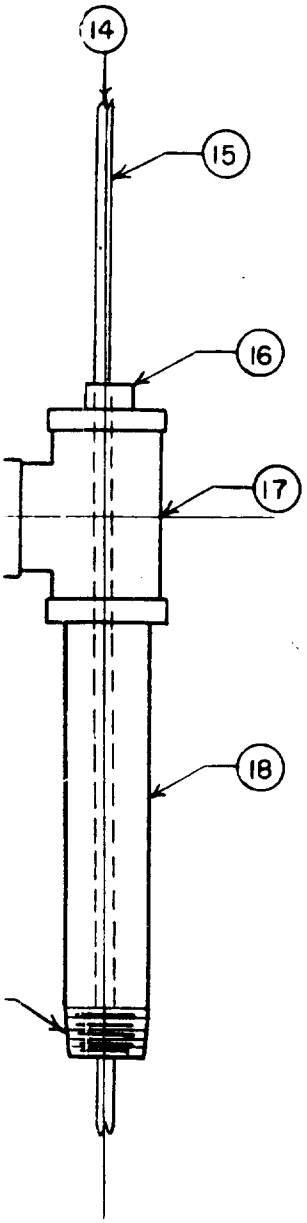

FIGURE 38

GAS-TIGHT ELECTRICAL TERMINAL HEAD FOR ELECTRICAL RESI



(1)

## LEGEND




1. $2\frac{1}{2}$" SECTION OF 3" STEEL PIPE
2. HERMETIC CURRENT TAP SEALS
3. HERMETIC THERMOCOUPLE SEALS
4. $\frac{1}{2}$" ANNULAR FLANGES $\frac{1}{8}$" STEEL PLATE, BRAZED
5. 6-32 X $\frac{1}{2}$" ST'D MACHINE SCREWS
6. FUSITE HERMETIC TERMINAL NO. A-2033 SOFT SOLDER
7. ST'D NEEDLE VALVE $\frac{1}{8}$" PIPE
8. 3" DISC OF $\frac{1}{16}$" BRASS
9. BRAZE
10. $\frac{1}{2}$ OF $\frac{1}{2}$" ST'D COUPLING
11. $\frac{1}{2}$" X 3" ST'D PIPE NIPPLE
12. $\frac{1}{2}$" ST'D PIPE UNION
13. $\frac{1}{2}$" X $2\frac{1}{2}$" ST'D PIPE NIPPLE
14. GAS INPUT
15. $\frac{3}{16}$" COPPER TUBE BRAZED IN PLUG EXTENDS TO CONTAINER BOTTOM
16. $\frac{1}{2}$" ST'D PIPE PLUG DRILLED $\frac{3}{16}$" HOLE
17. $\frac{1}{2}$" ST'D PIPE TEE
18. $\frac{1}{2}$" X $4\frac{1}{4}$" STD PIPE NIPPLE
19. SCREWS INTO CONTAINER TOP

SCALE: 1" = 2"

RE38

ECTRICAL RESISTIVITY SPECIMEN CONTAINER

introduced by silver soldering a length of 3/16" diameter copper tubing into the plug. The tubing was cut such that the length reached just to the bottom of the specimen container when the cover was securely bolted down. This arrangement allowed the flushing action of the protective atmosphere to sweep from the bottom to the top outlet of the specimen container.

The lateral outlet of the "tee" was fitted with a demountable pipe extension system composed of a 1/2 in. x 2 1/2 in. standard nipple, a 1/2 in. standard union, and a 1/2 in. x 3 in. standard nipple. Such a universal type coupling was considered necessary to facilitate placement of the circuit wires without unnecessary twisting when closing the pipe joints. The final nipple terminated in a gas-tight pot-head into which all electrical and thermocouple leads were led.

The pot-head was designed to be demountable in order to facilitate the ease of repair and modification. The unit was constructed from a section of 3 in. standard steel pipe, 2 1/2 in. long. Two steel annular flanges, 1/2 in. wide and 1/8 in. thick were brazed to the steel pipe. A pipe inlet from the container was made by drilling a 1/2 in. hole in the side of the pot-head and brazing a portion of a 1/2 in. standard pipe coupling in position. Cover plates for the faces of the pot-head, 3 in. in diameter, were machined from 1/16" hard-rolled brass sheet. These covers were centrally located on the annular flanges and were held in place by No. 6-32 x 1/2 in. long machine screws placed at intervals of 45°. The flanges were drilled and tapped to

receive the machine screws at the proper positions. Sealing between the cover plates and flanges was obtained by using 1/16 in. "Vellumoid"(a) sheet gasket material; washers under the cover screws aided in giving an even pressure distribution on the gasket over the flange area.

Gas-tight electrical terminals were obtained by using stock type hermetic seals recessed and soldered into each of the cover plates. Twelve outlet taps for the potential measuring circuit were located on the front cover plate through a 20 conductor "Fusite"(b) hermetic seal fitted with a keyed bakelite connector. This arrangement permitted ease of demounting the apparatus while maintaining identity of the measuring circuits. Current input and thermocouple leads were brought through four individual hollow tube hermetic seals on the back cover plate of the pot-head. The terminals were sealed to the cover plate by careful heating and allowing soft solder to fill the tube after the wire was projected through.

A gas escape mechanism on the side of the pot-head was provided in the form of an 1/8 in. brass needle valve fitted into a tapped hole. With this device, flushing action and rate of gas flow was observed by connecting the outlet side of the needle valve to a wash bottle.

A relatively low temperature was maintained at the pot-head to prevent damage to the hermetic seals. This was done by

(a) Mfg. by the Vellumoid Co., Worcester, Mass.
(b) Mfg. by the Fusite Corp., Cincinnati, Ohio.

a water cooling coil of 12 turns of 3/16 in. diameter copper tubing formed around the 2 1/2 in. length of pipe.

Revised Specimen Holder Assembly

A completely new specimen holding bracket was designed and built to provide a rugged and more easily used mount than the original holder. An assembly drawing of the improved specimen holder is shown in Fig. 39.

The foundation of each unit was a block of "Transite"(c) 1/2 in. x 1 in. x 1 1/4 in. A 5/32 in. groove was cut the entire length of each block to provide a uniform seat for the 1/8 in. diameter extruded rod specimen used in the determinations.

The electrical contacts to the specimens were made through two individual, insulated, brass attachments fitted to the transite block. Current is passed through the specimen ends by a small brass fitting with a hole concentric with the groove in the block and through which a brass thumb-screw applies positive pressure contact to the specimen. Refer to Fig. 39. The potential tap was made in the form of a sharply pointed and carburized No. 2-56 x 5/8 in. long machine screw projecting down through a small brass plate mounted directly over the specimen groove. Fastenings on the "Transite" blocks were made with No. 2-56 stainless steel machine screws.

The specimen mounts were fastened to the flat surfaces of a hexagonal, 2S aluminum block which was, in turn, securely fastened to the cover of the container to project 5 7/8 in. into

(c) Bonded Asbestos Sheet, Mfg. by Johns-Manville, 270 Madison Ave., New York, N.Y.

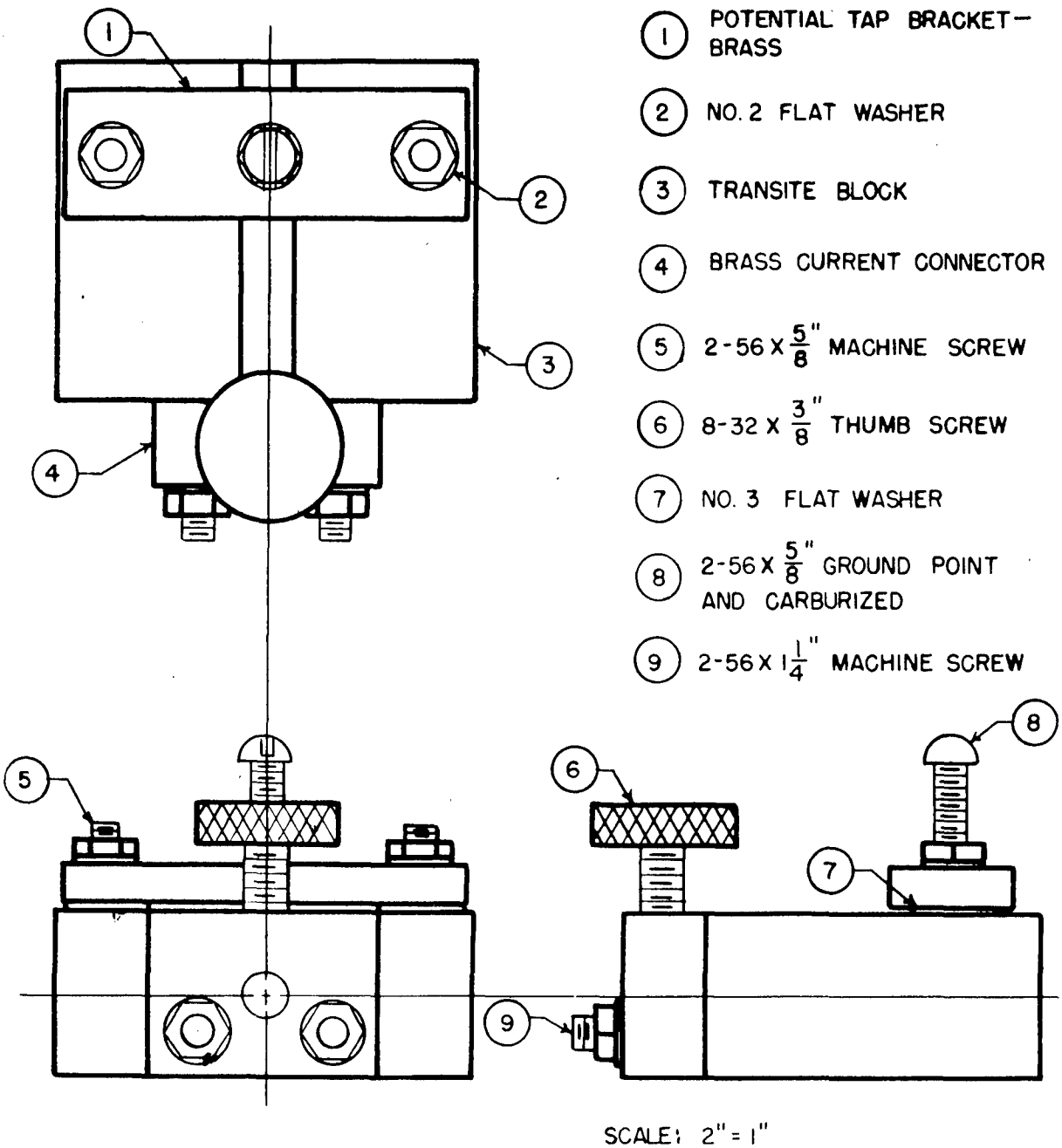



FIGURE 39

ASSEMBLED SPECIMEN HOLDER FOR ELECTRICAL RESISTIVITY APPARATUS. TWO HOLDERS PER SPECIMEN REQUIRED.

the container. Refer to Fig. 37. The specimen mounting blocks were fastened with No. 6-32 x 3/4 in. long machine screws to each of the sides of the hexagon at a carefully gauged length of 2 in. between potential taps. To assure complete insulation of the specimens from the support block, each mount was placed on a mica sheet which projected back sufficiently to shield the current connector from the aluminum base.

The current connectors were wired in series with 0.064 in. diameter solid copper wire fastened by washers under the heads of the current connector retaining screws. Two wires of 0.032 in. diameter solid copper were fastened to the potential tap bars, one to each end, and then led through "Transite" stand-off insulators to the pipe exit in the container cover.

Electrical insulation for the current and potential measuring circuits was secured by using "Varglas"(d) woven glass fibre tubing of proper wire size, capable of withstanding temperatures up to 1200°F. Additional protection was provided for the wires passing through the pipe exit system by wrapping the entire length with several turns of 1 1/2 in. woven asbestos tape.

A Chromel-Alumel thermocouple, for determining internal container temperature, was mounted on one of the Transite blocks and projected 3 3/4 in. down into the container. The couple is fibre-glass insulated with a porcelain tube at the welded end.

(d) Mfg. by Varflex Corp., Rome, N.Y.

## APPENDIX VI

## EXPERIMENTAL PROCEDURE FOR PREPARATION AND EVALUATION OF LOW ALLOY CONTENT MAGNESIUM BASE ALLOYS

### Melting and Alloying

All alloys were melted and alloyed in Tercod crucibles in a gas-fired furnace. Melt sizes averaged 3500 grams (7.7 pounds). The magnesium used was Dow Pure notched ingot. The charge was protected and refined with Dow 310 flux. The general procedure was to melt down the magnesium under flux, raise the melt temperature to 1350-1400°F, make all alloying additions at the same time as the element (or as discussed below) and immediately begin stirring for two minutes with a graphite or plain carbon steel rod. After the stirring operation any massive accumulation of flux on the melt surface was removed and fresh flux was sprinkled lightly on the surface. Melts were usually superheated to approximately 1500°F to permit better separation of non-metallics, removed from furnace, cooled to the pouring temperature range 1325-1350°F, and cast into ingot slabs for rolling into sheet. The temperature of the melting procedure in all phases was controlled by means of a chromel-alumel thermocouple immersed in the melt in a steel sheath. A spectrographic pencil was cast from each heat.

The melting and alloying procedures recommended by the Dow Chemical Company were followed as closely as possible. Specific details for alloying procedures used are summarized below.

1. Zinc was added as New Jersey Zinc Horsehead Special, 99.99 + % zinc.

2. Cerium was added as Cerium Standard, approximately 50% cerium, the balance being other rare earths and the order of 1% iron. The procedure followed was:

   a. Using Dow 310 flux, skim and sludge (if necessary) prior to making additions.

   b. Add Cerium Standard at 1350°F, together with other additions, if any, by placing in a hand ladle and washing gently below surface until dissolved.

   c. Stir thoroughly for at least two minutes and raise temperature of melt to 1450°F.

   d. Dust lightly with Dow 310 flux.

   e. Hold 15 minutes at 1450°F, remove from furnace and cool to 1350°F.

   f. Pour at 1350°F.

3. Zirconium was added either as (a) zirconium sponge or (b) dense zirconium tetrachloride.

   a. Based upon recommendations from the Dow Chemical Company, Magnesium Division, an experimental procedure was established for introducing the zirconium sponge in magnesium.

   A small quantity of zirconium sponge was obtained from the U.S. Bureau of Mines in the form of a compacted cake approximately 3 1/2 in. diameter and 2 in. thick. The cake was broken into small pieces

with a hammer and sized according to the following schedule:

| Size | Disposition |
|---|---|
| Greater than 1/2 in. | returned for further breakdown. |
| 1/2 in. to 1/8 in. | used for alloying in magnesium. This fraction was stored in a tightly sealed container. |
| Less than 1/8 in. | stored in a tightly sealed container and not used for alloying. |

The following procedure was followed in alloying magnesium with zirconium sponge:

1. Dow Pure was melted under Dow 310 flux and the melt temperature was raised to 1400°F and controlled at this temperature as closely as possible.
2. The sponge addition was weighed and placed in a cup-type ladle previously preheated to a red color. For a magnesium-1.0 zirconium composition a 3.2% addition of sponge was made.
3. The flux was pushed back, the ladle was inserted and was withdrawn immediately, holding a small quantity of melt in the cup. The cup was rested against the crucible wall above the melt and the melt helper stirred the tiny melt in this cup ladle with a pointed steel bar for approximately 30 seconds. The ladle was again

immersed to obtain a fresh portion of melt and the stirring was repeated. This cycle was continued until all of the sponge was removed from the cup. During this operation burning in the cup was controlled by quenching in the main melt, where Dow 310 flux was added when needed. An attempt was made to hold the melt temperature at 1400°F during alloying.

4. Following the introduction of zirconium, the melt was dusted lightly with Dow 310 and allowed to stand quietly for 15 minutes in the furnace at a temperature of 1400°F. This temperature was maintained as constant as possible.

5. The crucible was removed from the furnace, the melt was cooled and finally poured at 1350°F.

b. Addition of zirconium by means of dense zirconium tetrachloride.

The following general procedure was used in the addition of zirconium by means of dense zirconium tetrachloride:

1. Weigh the salt and then preheat it for two hours at 175-200°F wrapped in 0.002 in. aluminum foil.

2. Melt magnesium under Dow 310 flux and heat

to 1400 ± 25°F.

3. Skim the melt.
4. Immerse the foil-wrapped salt below the surface of the melt at 1400 ± 25°F, using Dow #310 liberally to control burning. Allow two minutes for bubbling condition to stop and then stir for three minutes.
5. Skim and dust with Dow #310 flux.
6. Hold the melt at 1400-1425°F for fifteen minutes to allow time for settling out of flux.
7. Remove from the furnace. Cool the melt to 1350°F and then pour it.

Two modifications of this method were made. The use of aluminum foil was discontinued as aluminum reduces the solubility of zirconium in magnesium. Also, preheating of the salt was omitted as unnecessary and perhaps detrimental. When the salt was preheated a distinct odor of hydrochloric acid was observed as well as a change in the surface appearance of the salt. The zirconium tetrachloride was stored in a sealed jar to prevent reactions with moisture in the air.

Casting Ingots for Rolling

Ingots for rolling were cast in a graphite mold in which two ingots were cast at one time from a single sprue using a large vertical riser over each ingot. The bottom surface of the ingots

cast in this mold became progressively roughened and porous so it was considered necessary to make a permanent cast iron mold. The principle of bottom feeding from a well of metal at one end of the ingots was incorporated.in the design of the new mold. In addition, a perforated steel screen was placed vertically in the sprue to screen out possible oxide or flux inclusions but this screen was discontinued after several trials. The surfaces of ingots cast in this iron mold were consistently better than those ingots cast in the graphite mold.

Rolling Procedure

The slabs were homogenized 16 to 20 hours at the temperature selected for hot rolling. All rolling was done on a 3 in. x 5 in. Oliver mill running at a constant speed of 16 1/2 feet per minute. The roll temperature was maintained constant at 400°F during all hot and warm rolling operations by means of gas flames manually regulated.

Warm rolling was carried out at 400°F in the case of magnesium-zinc-cerium alloys and at 500°F in the case of magnesium-zirconium alloys. Stock for warm rolling was hot rolled to 0.200 in., air cooled, and later heated to the warm rolling temperature.

In general, slabs were reduced to 0.200 in. on a schedule of constant draft. From 0.200 in. to 0.064 in., the slabs were reduced by a constant percentage of reduction between reheats. Sheet to be cold rolled was reheated one hour at the rolling temperature, air cooled, pickled and cold rolled 15% reduction at 0.001 in. per pass to the final thickness.

Thermal Treatment

Temperatures for annealing and stress relieving were determined from a recrystallization curve plotted for each condition of rolling for each composition. The heat treatment temperatures were selected to finish sheet in the following conditons:

1. "Stress-relieved" at a temperature near the lower limit of the recrystallization temperature range. Identified by "s" in alloy designations.
2. "Low temperature annealed" in the approximate middle of the recrystallization temperature range. Identified as "(L)" in alloy designations.
3. "High temperature annealed" near the upper limit of the recrystallization temperature range. Identified as "(H)" in alloy designations.

Thermal treatment after rolling was carried out on blanks sheared for later machining to test specimens. The blanks were compressed between flat plates during heating to flatten. Cold rolled blanks were flattened when necessary by heating between plates for 1 hour at 250 to 275°F.

Mechanical Testing

1. Hardness. Recrystallization curves were established by Vickers hardness testing equipment using a 5 kilogram load. Specimen preparation for hardness testing was carried through #3/0 metallographic emery paper.
2. Tensile Testing. Specimens for tensile testing were machined from blanks to a standard 2 in. gage length,

0.505 in. wide in a 3 in. long reduced section and 3/4 in. wide at the grip ends. The specimens were tested in a Southwark-Emery hydraulic testing machine using Templin Grips. Strain was measured with a Peters Extensometer in conjunction with a Southwark-Emery stress-strain recorder. The yield strength was taken at 0.2% offset on the stress-strain curve.

3. Compression Testing. Blanks were machined to 5/8 in. wide by 2 5/8 in. long specimens. These were mounted in a fixture to prevent buckling and tested in a Southwark-Emery hydraulic testing machine. Strain was measured with a Peters Averaging Compressometer in conjunction with a Southwark-Emery stress-strain recorder. The yield strength was taken at 0.2% offset on the stress-strain curve.

4. Notch Sensitivity Testing. Specimens used in the static notched bend test were 0.062 in. thick (data were corrected to this thickness value by means of a nomograph), 1 in. wide, 3 1/8 in. long, with a 45 degree notch in each edge located 3/4 in. from one end so that the distance between the roots of the notches was 0.50 in. The radius at the root of the notch was 0.030 in. Specimens were gripped in an Olsen Stiffness Tester such that the roots of the notches were 1/8 in. from the clamping bar of the vise. A load was applied 2 in. from the vise and simultaneous readings of percent of maximum bending

moment and angular deflection in degrees were recorded as the specimen was stressed to destruction. The following information was computed from the test data and tensile test data:

a. Ultimate Torque (in.-lbs.)

Ultimate Torque = % max. bend. moment x 40 in.-lbs.

b. Ultimate Bend Angle (Degrees); determined directly from test data.

c. Static Rupture Energy (in.-lbs.); obtained by integration of total area under percent maximum bending moment versus angular deflection curve with a planimeter.

d. $$\text{Strength Factor} = \frac{\text{Ultimate Torque (in.-lbs.)}}{\text{Ultimate Tensile Strength (kips per square inch)}}$$

e. $$\text{Ductility Factor} = \frac{\text{Ultimate Bend Angle (Degrees)}}{\text{Tensile \% Elongation in 2 inches}}$$

f. $$\text{Toughness Factor} = \frac{\text{Static Rupture Energy (in.-lbs.)}}{\text{Ultimate Tensile Strength x \% Elongation}}$$

Spectrographic Analyses

Spectrographic analyses, when determined, were made on a Baird 3 meter spectrograph in conjunction with a Leeds and Northrup Microphotometer and Speedomax Recorder. Specimens for analysis were 1/4 in. diameter pencils 4 in. long cast just prior to casting each heat.

0.505 in. wide in a 3 in. long reduced section and 3/4 in. wide at the grip ends. The specimens were tested in a Southwark-Emery hydraulic testing machine using Templin Grips. Strain was measured with a Peters Extensometer in conjunction with a Southwark-Emery stress-strain recorder. The yield strength was taken at 0.2% offset on the stress-strain curve.

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c. Static Rupture Energy (in.-lbs.); obtained by integration of total area under percent maximum bending moment versus angular deflection curve with a planimeter.

d. $$\text{Strength Factor} = \frac{\text{Ultimate Torque (in.-lbs.)}}{\text{Ultimate Tensile Strength (kips per square inch)}}$$

e. $$\text{Ductility Factor} = \frac{\text{Ultimate Bend Angle (Degrees)}}{\text{Tensile \% Elongation in 2 inches}}$$

f. $$\text{Toughness Factor} = \frac{\text{Static Rupture Energy (in.-lbs.)}}{\text{Ultimate Tensile Strength x \% Elongation}}$$

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# APPENDIX VII

## TABLE XX

## SUMMARY OF

## INTENDED COMPOSITIONS, SPECTROGRAPHIC ANALYSES AND MECHANICAL AND THERMAL TREATMENTS FOR LOW ALLOY CONTENT EXPERIMENTAL ALLOYS

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-703 | 0.5 | 0.5 | - | (a) | | | | | | | |
| R-704 | 0.5 | 0.5 | - | (a) | | | | | | | |
| 705 | 0.5 | 0.5 | - | (a) | | | | | | | |
| 706 | 1.0 | - | - | (a) | | | | | | | |
| 707 | 1.0 | - | - | (a) | | | | | | | |
| 708 | 1.0 | - | - | (a) | | | | | | | |
| 709 | 1.0 | - | - | (a) | | | | | | | |
| 710 | 1.0 | - | - | (a) | | | | | | | |
| 711 | 1.0 | - | - | (a) | | | | | | | |
| 712 | 1.0 | - | - | (a) | | | | | | | |
| 713 | 0.8 | 0.2 | - | (a) | | | | | | | |

(a) See notes at end of table.

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet Hot Rolled Stress Relieve | Low Temp. Anneal | Warm Rolled Stress Relieve | Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-714 | 0.8 | 0.2 | - | (a) | | | | | | | |
| 715 | 0.8 | 0.2 | - | (a) | | | | | | | |
| 716 | 0.9 | 0.1 | - | (a) | | | | | | | |
| 717 | 0.8 | 0.1 | - | (a) | | | | | | | |
| 718 | 0.9 | 0.1 | - | (a) | | | | | | | |
| 719 | This alloy number not used. | | | | | | | | | | |
| 720 | 1.0 | - | - | (a) | | | | | | | |
| 721 | 1.0 | - | - | (a) | | | | | | | |
| 722 | 1.0 | - | - | (a) | | | | | | | |
| 723 | 0.9 | 0.1 | - | (a) | | | | | | | |
| 724 | 0.9 | 0.1 | - | (a) | | | | | | | |
| 725 | 0.7 | 0.3 | - | (a) | | | | | | | |
| 726 | 1.0 | - | - | (a) | | | | | | | |
| 727 | 0.6 | - | - | (a) | | | | | | | |
| 728 | 0.7 (0.55) | - | - | 15 hr. | 700 | 700 | 400 | (b) | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-729 | 0.8 (0.64) | - | - | 15 hr. | 700 | 700 | 400 | (b) | | | |
| 730 | 0.6 (0.62) | 0.1 (0.09) | - | 15 | 700 | 700 | 400 | 500 | 750(c) | 400 | - |
| 731 | 0.6 (0.80) | 0.1 (0.11) | - | 15 | 700 | 700 | 400 | - | - | 400 | 700(c) |
| 732 | 0.7 | 0.1 | - | (a) | | | | | | | |
| 733 | 0.8 (0.80) | 0.1 (0.12) | - | (a) | | | | | | | |
| 734 | 0.6 | 0.2 | - | (a) | | | | | | | |
| 735 | 0.7 | 0.2 | - | (a) | | | | | | | |
| 736 | 0.8 | 0.2 | - | (a) | | | | | | | |
| 737 | 0.8 | 0.2 | - | (a) | | | | | | | |
| 738 | 0.8 (0.84) | 0.2 (0.16) | - | 15 hr. | 700 | 700 | 400 | 400 | 750(c) | 400 | 750(c) |
| 739 | 0.8 (0.90) | 0.2 (0.24) | - | 15 | 700 | 700 | 400 | 400 | 750(c) | 400 | 750(c) |
| 740 | 0.6 | 0.3 | - | (a) | | | | | | | |
| 741 | 0.7 | 0.3 | - | (a) | | | | | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp.°F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet Hot Rolled Stress Relieve | Low Temp. Anneal | Warm Rolled Stress Relieve | Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-742 | 0.8 (0.76) | 0.3 (0.31) | - | 15 hr. | 700 | 700 | 400 | 600 | 650 | 550 | 600 |
| 743 | 0.6 (0.62) | 0.5 (0.35) | - | 15 | 700 | 700 | 400 | 600 | 700 | 550 | 650 |
| 744 | 0.7 (0.68) | 0.5 (0.50) | - | 15 | 700 | 700 | 400 | 600 | 700 | 600 | 700 |
| 745 | 0.8 (0.74) | 0.5 (0.47) | - | 15 | 700 | 700 | 400 | 550 | 600 | 550 | 600 |
| 746 | 0.8 (0.68) | 0.2 (0.25) | - | (a) | | | | | | | |
| 747 | 0.7 (0.54) | 0.2 (0.23) | - | 15 | 700 | 700 | 400 | 600 | 750 | 500 | 600 |
| 748 | 0.6 | 0.2 | - | (a) | | | | | | | |
| 749 | 0.7 | 0.1 | - | (a) | | | | | | | |
| 750 | 0.6 (0.61) | - | - | 15 | 700 | 700 | 400 | - | - | (b) | |
| 751 | - | - | 0.1 (0.10) | 15 | 700 | 700 | 500 | - | - | (b) | |
| 752 | - | - | 0.1 (0.09) | 15 | 700 | 700 | 500 | - | - | (b) | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp.°F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-753 | - | - | 0.1 (0.20) | 15 hr. | 700 | 700 | 500 | - | - | (b) | |
| 754 | 0.6 (0.51) | 0.3 (0.37) | - | 15 | 700 | 700 | 400 | 600 | 650 | 550 | 600 |
| 755 | 0.7 (0.57) | 0.3 (0.28) | - | 15 | 700 | 700 | 400 | 600 | 650 | 550 | 600 |
| 756 | 0.8 (0.68) | 0.2 (0.22) | - | 15 | 700 | 700 | 400 | 700 | 750 | 450 | 600 |
| 757 | 0.8 (0.78) | 0.1 (0.21) | - | 15 | 700 | 700 | 400 | 400 | 650(c) | 400 | 600(c) |
| 758 | 0.6 (0.46) | 0.1 (0.20) | - | (a) | | | | | | | |
| 759 | 0.8 (0.78) | 0.2 (0.24) | - | 15 | 700 | 650 | 400 | 550 | 600 | 500 | 600 |
| 760 | 0.7 (0.53) | 0.1 (0.18) | - | 15 | 700 | 700 | 400 | 400 | 650(c) | 400 | - |
| 761 | 0.6 (0.46) | 0.2 (0.23) | - | 15 | 700 | 700 | 400 | 600 | 750 | 500 | 650 |
| 762 | - | - | 0.5 (<0.15) | 15 | 700 | 700 | 400 | - | - | (a) | - |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment to Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-763 | - | - | 1.0 (0.15-0.20) | 15 hr. | 700 | 700 | 400 | - | - | (d) | - |
| 764 | - | - | 0.5 (0.15-0.20) | 15 | 700 | 700 | 400 | - | - | (d) | - |
| 765 | - | - | 1.0 (0.4-0.45) | 15 | 700 | 700 | 400 | - | - | (d) | - |
| 766 | - | - | 0.5 (0.44) | 15 | 700 | 700 | 500 | 475 | 575 | 600 | 700 |
| 767 | - | - | 1.0 (0.73) | 15 | 700 | 700 | 500 | 600 | 750 | 500 | 700 |
| 768 | - | - | 0.1 (0.1-0.3) | (a) | | | | | | | |
| 769 | - | - | 1.0 (0.76) | 15 | 700 | 700 | 500 | 500 | 600 | 700 | - |
| 770 | - | - | 1.0 (0.54 | (a) | | | | | | | |
| 771 | - | - | 1.0 (0.59) | 15 | 700 | 700 | 500 | 450 | 600 | 550 | - |
| 772 | - | - | 1.0 (0.55) | 15 | 700 | 700 | 500 | 600 | 700 | 550 | 750 |
| 773 | - | - | 1.0 (0.60) | (a) | | | | | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-774 | 1.0 | 0.3 | - | (a) | | | | | | | |
| 775 | 1.0 | 0.3 | - | (a) | | | | | | | |
| 776 | 1.0 (1.0) | 0.3 (<0.1) | - | (a) | | | | | | | |
| 777 | 1.5 | 0.3 | - | (a) | | | | | | | |
| 778 | 1.5 (1.6) | 0.3 (0.13) | - | 15 hr. | 700 | 700 | 400 | - | - | 450 | 575 |
| 779 | 2.0 | 0.3 | - | (a) | | | | | | | |
| 780 | 2.5 | 0.3 | - | (a) | | | | | | | |
| 781 | 1.0 (1.0) | 0.3 (0.29) | - | 15 | 700 | 700 | 400 | 500 | 600 | 500 | 600 |
| 782 | 2.0 | 0.3 | - | (a) | | | | | | | |
| 783 | 2.0 (2.2) | 0.3 (0.20) | - | (a) | | | | | | | |
| 784 | 2.5 | 0.3 | - | (a) | | | | | | | |
| 785 | 3.0 (3.4) | 0.3 (0.40) | - | (a) | | | | | | | |
| 786 | 2.0 (2.2) | 0.3 (0.31) | - | (a) | | | | | | | |
| 787 | 2.5 (2.9) | 0.3 (0.58) | - | (a) | | | | | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp.°F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment of Sheet Hot Rolled Stress Relieve | Low Temp. Anneal | Warm Rolled Stress Relieve | Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-788 | 0.8 (0.91) | 0.3 (0.40) | - | 15 hr. | 700 | 700 | 400 | 450 | 575 | 400 | 450 |
| 789 | 0.8 (1.0) | 0.3 (0.27) | - | 15 | 700 | 700 | 400 | 325 | 450 | 400 | 550 |
| 790 | 0.8 (0.80) | 0.3 (0.30) | - | 15 | 700 | 700 | 400 | (e) | | | |
| 791 | 0.8 (0.9) | 0.3 (0.15) | - | 15 | 700 | 700 | 400 | 600 | 675 | 450 | 550 |
| 792 | 1.5 (1.5) | 0.3 (0.15) | - | (a) | | | | | | | |
| 793 | 0.8 (0.8) | 0.3 (0.15) | - | 15 | 700 | 700 | 400 | 450 | 575 | 300 | 575 |
| 794 | 1.5 (1.4) | 0.6 (0.37) | - | 15 | 700 | 700 | 400 | 575 | 700 | 350 | 500 |
| 795 | 2.0 (2.3) | 0.8 (0.37) | - | (a) | | | | | | | |
| 796 | 2.5 (2.7) | 1.0 (0.57) | - | 15 | 700 | 700 | 400 | 475 | 550 | 300 | 425 |
| 797 | 3.0 (3.3) | 1.2 (0.95) | - | (a) | | | | | | | |
| 798 | 3.0 (3.3) | 0.3 (0.18) | - | 15 | 700 | 700 | 400 | 500 | 550 | 350 | 475 |
| 799 | - | - | 0.3 (0.67) | (a) | | | | | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment to Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-800 | 0.2 (0.33) | - | 0.8 (0.83) | (a) | | | | | | | |
| 801 | 0.5 | - | 0.5 | (a) | | | | | | | |
| 802 | 0.8 | - | 0.2 | (a) | | | | | | | |
| 803 | 0.2 | - | 0.8 | (a) | | | | | | | |
| 804 | 0.8 | - | 0.2 | (a) | | | | | | | |
| 805 | 1.5 | 0.6 | - | (a) | | | | | | | |
| 806 | 2.0 | 0.8 | - | (a) | | | | | | | |
| 807 | 1.5 (1.40) | 0.6 (0.43) | - | 15 hr. | 700 | 700 | 400 | (b) | - | 400 | 525 |
| 808 | 2.0 (2.15) | 0.8 (0.64) | - | 15 | 700 | 700 | 400 | 375 | 475 | 375 | 500 |
| 809 | 2.5 (2.5) | 1.0 (0.55) | - | 15 | 700 | 700 | 400 | 400 | 450 | 375 | 500 |
| 810 | 1.5 (1.2) | 0.6 (0.31) | - | 15 | 700 | 700 | 400 | 550 | 625 | 350 | 550 |
| 811 | 2.0 (1.65) | 0.8 (0.41) | - | 15 | 700 | 700 | 400 | 425 | 525 | 450 | 600 |
| 812 | 2.5 (2.3) | 1.0 (1.10) | - | 15 | 700 | 700 | 400 | 500 | 575 | 475 | 600 |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment to Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-813 | 3.0 (2.5) | 1.2 (0.37) | - | 15 hr. | 700 | 700 | 400 | 425 | 525 | 325 | 450 |
| 814 | 1.5 (1.2) | 0.3 (0.38) | - | 15 | 700 | 700 | 400 | 500 | 650 | 450 | 575 |
| 815 | 2.0 (2.0) | 0.3 (0.31) | - | 15 | 700 | 700 | 400 | 375 | 525 | 350 | 525 |
| 816 | 2.5 | 0.3 | - | (a) | | | | | | | |
| 817 | 3.0 (3.2) | 0.3 (0.31) | - | 15 | 700 | 700 | 400 | 400 | 525 | 325 | 475 |
| 818 | - | 1.0 | - | (a) | | | | | | | |
| 819 | - | 1.3 | - | (a) | | | | | | | |
| 820 | - | 1.0 | - | (a) | | | | | | | |
| 821 | - | 1.6 | - | (a) | | | | | | | |
| 822 | 2.5 (2.8) | 0.3 (0.40) | - | 15 | 700 | 700 | 400 | 350 | 450 | 375 | 500 |
| 823 | 3.0 (3.2) | 0.3 (0.34) | - | 15 | 700 | 700 | 400 | 350 | 475 | 375 | 500 |
| 824 | 3.5 (3.4) | 0.3 (0.39) | - | 15 | 700 | 700 | 400 | 350 | 500 | 325 | 400 |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment to Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-825 | - | 1.0 | - | (a) | | | | | | | |
| 826 | - | 1.3 | - | (a) | | | | | | | |
| 827 | - | 1.6 | - | (f) | | | | | | | |
| 828 | - | 1.9 | - | (a) | | | | | | | |
| 829 | - | 2.2 | - | (a) | | | | | | | |
| 830 | - | 2.5 | - | (a) | | | | | | | |
| 831 | - | 0.9 | - | (f) | | | | | | | |
| 832 | - | 0.6 | - | (f) | | | | | | | |
| 833 | - | 1.0 | - | (f) | | | | | | | |
| 834 | 3.5 | 0.3 | - | (e) | | | | | | | |
| 835 | 0.8 (0.76) | 0.3 (0.23) | - | (a) | | | | | | | |
| 836 | 0.8 (0.77) | 0.3 (0.22) | - | (a) | | | | | | | |
| 837 | 0.8 (0.78) | 0.3 (0.27) | - | (a) | | | | | | | |
| 838 | 0.8 (0.74) | 0.3 (0.24) | - | (a) | | | | | | | |

TABLE XX, Cont'd

| Alloy | Intended Compositions & Spectro. Anal., Weight Percent Zn | Ce | Zr | Ingot Homogenization Prior to Rolling Time | Temp. °F | Nominal Rolling Temp. °F Hot | Warm | Thermal Treatment to Sheet: Hot Rolled Stress Relieve | Hot Rolled Low Temp. Anneal | Warm Rolled Stress Relieve | Warm Rolled Low Temp. Anneal |
|---|---|---|---|---|---|---|---|---|---|---|---|
| R-839 | 0.8 (0.74) | 0.3 (0.15) | - | | (a) | | | | | | |
| 840 | 0.8 (0.58) | 0.3 (0.23) | - | | (a) | | | | | | |
| 841 | 0.8 (0.50) | 0.3 (0.17) | - | | (a) | | | | | | |
| 842 | 0.8 | 0.3 | - | | (a) | | | | | | |
| 843 | 0.8 (0.51) | 0.3 (0.15) | - | | (a) | | | | | | |
| 844 | 0.8 (0.44) | 0.3 (0.21) | - | | (a) | | | | | | |
| 845 | 0.8 (0.45) | 0.3 (0.16) | - | | (a) | | | | | | |

(a) Ingots were scrapped because of poor quality or because of alloy off-compositions.
(b) Specimens were tested in the as-rolled condition.
(c) High temperature anneal was used.
(d) Ingots were used for practice rolling.
(e) Sheet is stored for future use.
(f) Alloy was used for a cerium spectrographic standard.