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# PROPERTIES OF SAND-CAST MAGNESIUM ALLOYS

**PART I:** Binary Magnesium-Zinc Alloys

B. LAGOWSKI & J. W. MEIER

PHYSICAL METALLURGY DIVISION

JUNE 1958

**RELEASED FOR GENERAL DISTRIBUTION - 1964** 

EPARTMENT OF MINES AND CHNICAL SURVEYS, OTTAWA

MINES BRANCH

RESEARCH REPORT

R 9

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Catalogue No. M38-1/9

Price subject to change without notice

ROGER DUHAMEL, F.R.S.C. Queen's Printer and Controller of Stationery Ottawa, Canada 1965

# FOREWORD

After the end of World War II, in 1945, the Canadian Bureau of Mines (at present the Mines Branch) established an extensive research program in the field of magnesium and magnesium alloy technology. The aims of this endeavour were

- (a) to help the newly created Canadian magnesium industry to survive the conversion to peace-time conditions and to establish a broader base for the inclusion of this new industrial metal in the Canadian economy; and
- (b) to assist the Canadian Armed Forces in the development of materials of high strength-toweight ratio for use in aircraft and airborne equipment.

A considerable part of this research effort was directed to developing highest-strength magnesium casting alloys. Some of the results of these studies, especially the development of magnesium casting alloy ZK61, have been presented in various technical papers, the first of which appeared in 1948. The present series of reports covers the results of some of the other investigations which are of more general interest.

ohn Convey

Director, Mines Branch

#### Mines Branch Research Report R 9

# PROPERTIES OF SAND-CAST MAGNESIUM ALLOYS.

# Part I: Binary Magnesium-Zinc Alloys

by

B. Lagowski\* and J. W. Meier\*\*

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## ABSTRACT

The magnesium-zinc alloy system is today the most important base for high-strength magnesium alloys. Unfortunately, published data on properties of binary Mg-Zn casting alloys are both limited and controversial. It was, therefore, necessary to undertake a systematic study of the effect of composition and various melt treatments on the foundry characteristics, amenability to heat treatment, mechanical properties, and metallographic structure of this alloy series. Work on grain refinement of magnesiumzinc alloys included superheating, carbon inoculation, and additions of small amounts of aluminum, iron, manganese, titanium and zirconium. Results of the investigation show the necessity for considerable revision of long accepted data.

\* Scientific Officer and \*\* Principal Metallurgist (Non-Ferrous Metals), Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

# Direction des mines

#### Rapport de recherches R 9

# PROPRIÉTÉS DES ALLIAGES DE MAGNÉSIUM COULÉS EN SABLE

# Partie I: Alliages binaires de magnésium et de zinc

par

B. Lagowski\* et J. W. Meier\*\*

résumé

Le système des alliages magnésium-zinc est actuellement à la base de la plupart des alliages de magnésium à haute résistance. Les renseignements publiés, concernant les propriétés des pièces moulées d'alliages binaires Mg-Zn, sont malheureusement à la fois restreints et sujets à controverses. C'est pourquoi il a été nécessaire d'entreprendre une étude systématique de l'effet de la composition chimique et des divers traitements du bain sur les caractéristiques de fonderie, l'aptitude aux traitements thermiques, les propriétés mécaniques, et la structure métallographique de cette série d'alliages. Les travaux relatifs à l'affinage de grain des alliages de magnésium-zinc comprennent le surchauffe, l'ensemencement au carbone et l'introduction de faibles quantités d'aluminium, de fer, de manganèse, de titane et de zirconium. Les résultats de ces recherches démontrent la nécessité de reviser à fond les données admises depuis longtemps.

\*Agent scientifique et \*\*métallurgiste principal (métaux non ferreux), Division de la métallurgie physique, Direction des mines, ministère des Mines et des Relevés techniques, Ottawa, Canada.

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# INTRODUCTION

Zinc has been used for a long time in commercial magnesium alloys, but it is less than twenty-five years since it became the major alloying addition in the modern high-strength alloys containing zirconium, rare earths, or thorium. It is in connection with the development of these more complex alloy systems that a review of the properties of the binary magnesium-zinc alloys has been included in the broad research program on high-strength magnesium casting alloys, conducted over the past decade<sup>(1)</sup> at the Physical Metallurgy Division of the Mines Branch, Department of Mines and Technical Surveys, in Ottawa.

## LITERATURE SURVEY

The most recent work on the equilibrium diagram of magnesiumzinc alloys was published by Anderko et al<sup>(2)</sup>. A short discussion of this diagram and of the various Mg-Zn alloy phases found in the as-cast and heat treated alloys is given in Part II of this report series<sup>(3)</sup>.

Published data on mechanical properties of sand-cast magnesiumzinc alloys were found to be limited and controversial, Maybrey<sup>(4)</sup> reported an increase of mechanical properties of a sand-cast Mg-8Zn alloy by bottom pouring and still higher properties in chill castings.

The effect of zinc content on ultimate strength and density of sandcast binary Mg-Zn alloys with 4-12% Zn was first reported by Aitchinson<sup>(5)</sup>, according to whom the maximum tensile strength was obtained at 8% Zn.

Stoughton and Miyake<sup>(6)</sup> found that Mg-Zn alloys could be hardened by heat treatment. Gann and Winston<sup>(7)</sup> investigated the effect of zinc on tensile strength, impact toughness and Brinell hardness of sand-cast binary Mg-Zn alloys, in the as-cast condition, obtaining maximum tensile strength at approximately 6% Zn. Gann<sup>(8)</sup> checked hardness of binary Mg-Zn alloys after various heat treatments and found the highest values, after solution heat treating, quenching, and ageing, in the alloy with highest zinc content.

Dumas and Rockaert<sup>(9)</sup> investigated the relation between tensile strength, elongation and Brinell hardness, and the zinc content in sandcast and extruded binary Mg-Zn alloys. In sand-cast alloys they obtained a maximum tensile strength at approximately 6% Zn, and a steady decrease of elongation with increasing zinc contents.

Haughton and Prytherch<sup>(10)</sup> report results obtained at the National Physical Laboratory, which show a maximum tensile strength at 4% Zn in the as-cast condition and at 5% Zn in the fully heat treated condition, while elongation decreases in both conditions with increased zinc content.

Beck<sup>(11)</sup> quotes the results of P. Spitaler who obtained maximum values in tensile strength and elongation at approximately 5% Zn.

Fox(12) found that maximum UTS in binary Mg-Zn casting alloys is obtained with 5% Zn in the as-cast condition and 4% Zn in the fully heat treated condition. The elongation decreases in the as-cast and reaches maximum at 2% Zn in the fully heat treated condition; and 0.1% yield strength increases in both conditions with increased zinc content. He also states that binary Mg-Zn alloys with 4 and 6% Zn are very prone to microporosity.

Busk and Marande<sup>(13)</sup> prepared a series of binary Mg-Zn alloys from commercial magnesium containing 0.1 to 0.2% Mn; all alloys were superheated. The UTS reaches a maximum at approximately 6% Zn in both as-cast and fully heat treated conditions, and the elongation reaches a maximum at 2% Zn in both conditions.

Leontis<sup>(14)</sup> investigated mechanical properties of sand-cast binary Mg-Zn alloys in the range 0 to 10% Zn, prepared from electrolytic magnesium containing 0.03% Fe and 0.05 to 0.1% Mn. In the as-cast condition he found the highest tensile strength in alloys with 2 to 6.5% Zn, the yield strength increasing with the zinc content, and an elongation maximum at 2% Zn. In the heat treated conditions, both ultimate tensile and yield strength rise progressively with the zinc content and the maximum elongation values are at 1% in the solution heat treated, and at 2% Zn in the fully heat treated condition. Meier<sup>(15)</sup> in his discussion of this paper presented data on the effect of zinc on mechanical properties (UTS and 0.2% YS) of sand-cast binary Mg-Zn alloys containing up to 11% Zn in the as-cast and fully heat treated conditions. In both conditions, UTS and 0.2% YS increase with the zinc content. The results show, in both conditions, some anomalous decrease in UTS values in the range of approximately 4-8% Zn.

The effects of zinc content on the mechanical properties of sandcast binary Mg-Zn alloys in the as-cast condition, as reported by various investigators, are summarized graphically in Figure 1. Most of the curves for the ultimate tensile strength show a maximum for zinc contents ranging from 4 to 8% Zn, followed by a sharp decrease, with the exception of the earlier results of work at these laboratories, reported by Meier(15) which show that the tensile strength increases progressively with the zinc content. Yield strength curves show in all cases a steady increase with rising zinc content. Elongation curves show a steady decrease, with the exception of two cases where a maximum value is shown at 2% and 5% respectively. For comparison purposes, the results of the present investigation are also shown in Figure 1.

Similarly, the results of various investigators for alloys in the fully heat treated condition are presented in Figure 2. Here, again, some of the ultimate tensile strength curves reach a maximum at 4 to 6% Zn followed by a rapid decrease, with the exception of the results given by Leontis<sup>(14)</sup> and Meier<sup>(15)</sup> which show a progressive increase of strength with rising zinc content. The character of the yield strength and elongation curves is generally the same.

It must be appreciated that in most of the foregoing investigations the materials used were of rather low purity and that, at least in the earlier studies, modern melting, refining and casting procedures were unknown.

In view of the disagreement in published data, it was considered desirable to undertake a systematic study of the effects of composition and various melt treatments on foundry characteristics, amenability to heat treatment, mechanical properties, and metallographic structure of sand-cast magnesium-zinc alloys.

As may be seen from the graphs in Figures 1 and 2, the results obtained in this investigation differ considerably from those published by earlier workers.

#### MATERIALS AND EXPERIMENTAL PROCEDURES

#### Materials

Materials used for preparation of the alloys included Domal 99.98% Mg ingots and Tadanac 99.99% Zn ingots. The melts were prepared in welded steel pots of 40 lb capacity, using Domal crucible flux<sup>(16)</sup>.

Test bars<sup>\*</sup> were cast-to-shape (Figure 3) in green sand, using standard sand practice for magnesium alloys (16), and were tested without machining.

\* Test bar design according to Canadian Draft Standard CSA.HG.1-1956 and U.S. Federal Specification QQ-M-56, Figure 1A.

#### Melting Procedure

In earlier work conducted at these laboratories on binary sandcast magnesium-zinc alloys, erratic tensile properties had been obtained on alloys containing approximately 3 to 9% Zn. In order to establish a melting procedure that would consistently give optimum mechanical properties, a series of melts of Mg-6% Zn alloy was prepared on a laboratory scale (25 lb) and various melting variables were investigated. The results of these tests indicated that the most consistent improvement in metal quality was achieved when:

- (a) the metal was poured at the lowest possible temperature;
- (b) the flux and the metal were preheated at 400 °C (750 °F) for at least four hours for the former and two hours for the latter; and
- (c) the melt was settled for a prolonged period of time (20-60 min) at the lowest possible temperature above liquidus.

These preliminary results also indicated that superheating was detrimental (possibly due to gas absorption and/or iron pick-up). An attempt to grain-refine by either superheating or carbon inoculation was unsuccessful. However, when electrolytic (lower purity) magnesium was used, a slight improvement in mechanical properties -- still without any appreciable change in grain size -- was obtained after carbon inoculation.

Based on the above results, the following melting procedure was established: The steel pot (capacity, 40 lb magnesium) was preheated in a gas-fired furnace, cleaned, and sprinkled with preheated (4 hr at 400 °C) crucible flux. Preheated (2 hr at 400 °C) magnesium ingots were charged into the pot and melted under crucible flux. When all magnesium was molten, the temperature was raised to 690 °C (1275 °F). Zinc metal was added, the melt was stirred for three minutes with a steel spoon, the temperature was dropped to approximately  $10 \,^{\circ}C$  (18  $^{\circ}F$ ) above the liquidus, and, for alloys with 0-1.5% Zn, the melt was held at that temperature for twenty minutes. This settling time was increased by five minutes for each additional 0.5% Zn, until a maximum of forty-five minutes was reached for alloys containing from 4 to 30% Zn. At the end of the settling period the temperature of the melt was raised to approximately 50 °C (90 °F) above the liquidus and eight sets of green sand moulds, yielding 32 cast-to-shape test bars (Figure 3), were poured. Shake-out time was, in all cases, 35 minutes after the end of pouring.

#### Heat Treatment

Solution heat treatments were carried out in an electricallyheated circulating air furnace with an automatically-controlled protective atmosphere containing 1% SO<sub>2</sub>; the temperature was controlled to  $\frac{1}{2}$  °C. Ageing was carried out in an electric oven with air circulation and close temperature control (to  $\frac{1}{2}$  1 °C).

#### **Testing** Procedure

All test bars were radiographed and four unmachined test bars were tested at room temperature in the as-cast condition within 72 hours from the time of casting (to avoid the effects of room temperature ageing). Samples for chemical analysis were drilled from the grip end of one-half of the broken test bars, and the other half was used for the determination of density, grain size, and hardness (Rockwell "E" scale). Approximately two weeks, and three, six and twelve months after the testing of the first four test bars of each melt, two more test bars of each melt were tested to check the effect of room-temperature ageing on mechanical properties. Two test bars from each composition were tensile-tested at room temperature in the solution heat treated (T4), aged (T5), and fully heat treated (T6) conditions. One test bar from each alloy was selected for more detailed metallographic study.

## Alloy and Temper Designations.

Alloy and temper designations used throughout this report are according to Canadian standards CSA.H.1.1.-1958 (alloy designations) and CSA.H.1.2.-1958 (temper designations). The letter Z in the alloy designation indicates zinc, and the number following it represents the percentage of the alloy content; for example "Z6" designates a binary Mg-6% Zn alloy. In the temper designations, "F" designates the "as-fabricated" ("as-cast"), "T4" the solution heat treated, "T5" the artificially aged only, and "T6" the solution heat treated and artificially aged condition.

#### Test Results

For reasons of clarity, test results are presented in this report as graphs only; the detailed results obtained for each alloy and property are listed in Physical Metallurgy Division Internal Report PM-R-58-8 (1958).

#### PROPERTIES IN THE AS-CAST CONDITION

# Tensile Properties

Tensile test results obtained in this investigation on binary alloys with up to 30% Zn are presented in more detail in Figure 4. For alloys containing from 3 to 9% Zn, the graph shows a considerable drop in ultimate tensile strength and elongation, which does not conform to the general trend indicated by the results obtained on the rest of the alloys tested. This anomalous behaviour was considered spurious, especially since both yield strength and hardness (see Figure 9) showed no irregularities in this zinc range.

X-ray examination of test bars containing 3 to 9% Zn revealed varying amounts of scattered microporosity in the gauge lengths and, in addition, concentrated microporosity in planes perpendicular to the axis of the test bar. This layer porosity, as shown in Figure 5, has been investigated in Mg-Al and Mg-Zn alloys, and a discussion of its origin and occurrence is given by Baker(17).

Figure 6 shows the layer porosity in the gauge length of a test bar as revealed by X-ray examination. It is evident that this type of defect may decrease the UTS and elongation values considerably.

Test bars cast in alloys containing less than 3%, or more than 9% Zn, showed no microshrinkage. It was therefore assumed that tensile results on "sound" (porosity-free) test bars of alloys containing 3 to 9% Zn should follow the solid lines of the graph (Figure 4). To produce such porosity-free bars would have required considerable development work to improve the melt quality by special refining treatments or to redesign gating and risering of the test bar castings. To avoid this, it was decided to check the true properties of sound metal of this composition range in another way. Each test bar was radiographed (Figure 6), a sound portion of the gauge length was notched, as shown in Figure 7, and the notched bar was tensile-tested.

Figure 8 presents the results of these tensile tests, which follow in a surprisingly regular way the trend of the original curve. The difference in properties shown by the two curves is due to the stress-raising effect of the notch<sup>(18)</sup> -- this was confirmed by additional tests on unnotched and notched specimens, taken from entirely sound material from alloys containing less than 2% or more than 10% Zn. The higher values for UTS - 7 - -

and the lower values for elongation, obtained on notched test bars, were checked also on extruded Mg-3% Zn alloy, and showed the same effect of notching. Timoshenko(18) explains that "... in testing specimens with deep grooves, a certain increase in the ultimate strength is usually obtained because the grooves prevent necking of the specimen at the cross-section of the fracture".

# Hardness, Density, and Grain Size

The results of hardness, density and grain size measurements are presented in Figure 9 and show a steady increase of hardness and density values with increasing zinc content. The grain size curve shows an initial rapid decrease, levelling off at approximately 15% Zn. Higher values obtained on alloys containing more than 15% Zn are due to difficulties in the determination of grain size and are represented by a dotted line.

# FOUNDRY CHARACTERISTICS

Figure 10 presents the effect of zinc content on the linear pattern shrinkage. It may be of some interest to note that the shape of the curve closely follows the relationship between zinc content and hot tearing characteristics, reported in an earlier publication (19) and reproduced in Figure 11; the peak of the curve occurs around 1% Zn in both cases.

#### HEAT TREATMENT

The next problem was heat treatment. As shown in Figure 12 solution heat treating temperatures and times were chosen arbitrarily in relation to the solidus line<sup>(20)</sup> for this part of the alloy system. The solution temperatures were:  $520 \,^{\circ}\text{C}$  (970 °F) for alloys containing up to 3% Zn, decreasing gradually to 330 °C (625 °F) for alloys of 8% Zn and above. Heat treating times were varied from two and one-half hours at the highest temperature to sixteen hours at the lowest temperature, as indicated on the graph.

Ageing treatment was the same for all alloys and consisted of heating the bars for sixteen hours at  $180 \,^{\circ}\text{C}$  (355  $^{\circ}\text{F}$ ).

Figure 13 presents the effect of various heat treatments on the ultimate tensile strength of the alloys. It should be added here that some results in the range of 3 to 9% Zn are based on corrected values checked on notched bars, as described for the as-cast condition.

The marked decrease of the ultimate tensile strength in the T4 condition of alloys containing over 6% Zn, coincides with the appearance of a second phase which, in this temper, is the  $Mg_7Zn_3$  compound. In the other tempers, this second phase appears as an eutectoid of magnesium solid solution and MgZn compound, as shown in Part II of this report series<sup>(3)</sup>.

Figure 14 shows similar curves for the 0.2% yield strength. The character of the curves follows closely that of the curves for hardness in the same conditions. Figure 15 presents the elongation values, which attain a maximum at approximately 2% Zn regardless of condition. Figure 16 shows the hardness and grain size values after heat treatment.

# ROOM TEMPERATURE AGEING

In previous publications<sup>(21,22)</sup> it was reported that, after being stored for extended periods, Mg-Zn and Mg-Zn-Zr alloys showed significant increases in yield strength and hardness, as well as somewhat smaller increases of the ultimate tensile strength. This effect of room temperature ageing was also found, in the present investigation, on the binary Mg-Zn alloys containing more than 3% Zn. Properties of as-cast test bars after room temperature ageing for two weeks, and for three, six and twelve months, were determined.

Results of the investigation showed that no change of properties could be found after two weeks of storing. Some increase of yield strength was noted after three months, for alloys containing 6% and more zinc. Figure 17 shows the results after six and twelve months' ageing. Both the ultimate tensile strength and, especially, the yield strength increased considerably for alloys containing more than 4% Zn, whereas the elongation decreased slightly.

Test bars left over from an earlier investigation and tested after five years in storage showed further increases in both the UTS and YS, with a further slight decrease in elongation.

#### MICROSTRUCTURE

Figure 18 shows the equilibrium diagram of the binary Mg-Zn system as revised by Anderko et al<sup>(2)</sup>. The most interesting points are the formation of the eutectic of magnesium solid solution and  $Mg_7Zn_3$  compound at 343 °C (650 °F), and the eutectoidal decomposition of the  $Mg_7Zn_3$  compound into magnesium solid solution and MgZn compound at approximately 330 °C (626 °F).

A more detailed description of the effect of various heat treating conditions on the microstructure of binary Mg-Zn alloys is given in Part II of this report series<sup>(3)</sup>.

#### GRAIN REFINEMENT

Further improvement of properties was sought by grain refinement. Work on zirconium additions to Mg-Zn alloys showed that effective grain refinement coincidentally eliminates the layer-type porosity, discussed earlier, that was found in the gauge length of test bars.

Grain-size behaviour in cast magnesium alloys and also various methods of effective grain refining were discussed in great detail by C. E. Nelson<sup>(23)</sup>. Unfortunately, most of the grain-refining methods (superheating, treatments by chlorine or chlorine and carbon compounds) are ineffective for magnesium-zinc alloys and, according to Siebel<sup>(24)</sup>, may even cause grain coarsening. The only method he found to produce some grain refinement in these alloys was the German Elfinal (ferric chloride) process. Mannchen<sup>(25)</sup> proposed introduction of hydrogen, or hydride-forming elements, into the melt as grain refiners.

As already mentioned, earlier in the present investigation it was also found that superheating was detrimental and carbon inoculation ineffective. It was therefore decided to undertake an exploratory study of the effect of various metallic additions on the grain size and tensile properties of sand-cast magnesium-zinc alloys.

#### Zirconium Additions

Work on zirconium additions to magnesium and magnesium-zinc casting alloys, carried out at these laboratories, was reported earlier(21,22) and is summarized in Figures 19 and 20. The curves in Figure 19 show(22) the considerable influence of high soluble (or effective) zirconium content on grain size and tensile properties of magnesium alloy Z6 (Mg-6% Zn). The effect of zinc content on properties of as-cast and heat-treated Mg-Zn-Zr casting alloys is presented in Figure 20, showing maximum properties for alloy ZK61(22).

It may be observed that zirconium additions, introduced first in Germany in 1938, are used at present in all commercially established alloys based on the Mg-Zn, Mg-R.E., Mg-Th, Mg-Zn-R.E. and Mg-Zn-Th systems.

#### **Titanium Additions**

The considerable success in the use of zirconium in the grain refinement of magnesium and magnesium alloys prompted a parallel study of titanium additions. A detailed description of this investigation will be published in a separate report. The general results of the effect of small titanium additions are included in Figures 21 and 22.

#### Other Additions

Other elements added as possible grain refiners or to eliminate layer porosity were: A1, Bi, Cd, Li, Mn, Mn + Fe, Pb, R.E., Sn, and Th, in amounts of 0.5% to 1% (with the exception of A1, which was added up to 2%). The most successful were the additions of 1.5% A1 with carbon inoculation, and 0.7% Mn + 0.06% Fe. Tensile properties of as-cast Mg-Zn alloys are shown in Figure 21 and compared with those obtained on alloys with various grain refining additions. It should be noted that manganese additions decrease the properties of the binary alloys, while Mn + Fe additions act as a grain refiner and cause a general increase of properties, particularly at lower zinc contents. The elimination of layer-type porosity was achieved only by addition of the elements which were effective grain refiners. Figure 22 shows the effect of the various grain-refining additions on mechanical properties of Mg-Zn alloys in the fully heat treated (T6) condition.

#### CONCLUSIONS

The limited information which has been published on the relationship between the alloy composition and mechanical properties of binary Mg-Zn casting alloys is very controversial. The present investigation shows that alloys in the range of 3 to 9% Zn are affected by layer porosity and, therefore, do not show the "true" tensile properties, which can be revealed by the use of notched test bars. Grain-refining additions (Zr, Ti, Al + carbon inoculation, Mn + Fe) improve mechanical properties of the alloys and eliminate the layer-type porosity in the test bar casting.

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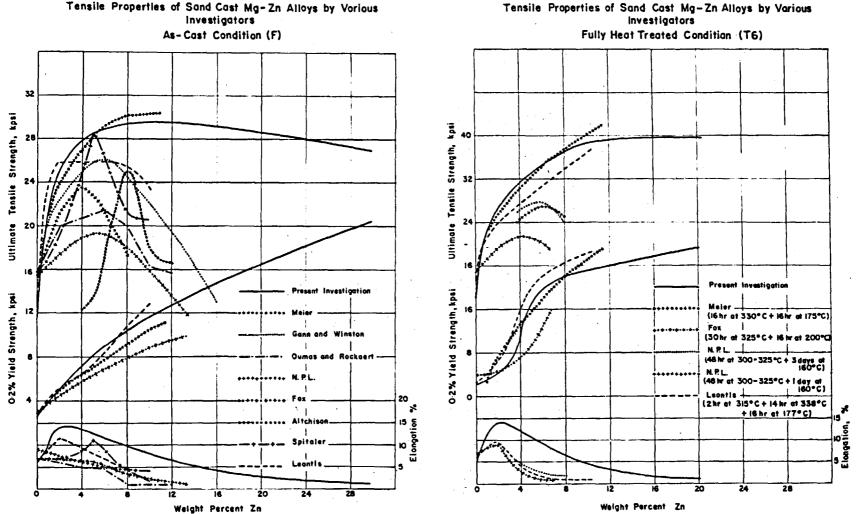
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Tensile Properties of Sand Cast Mg-Zn Alloys by Vorious

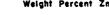
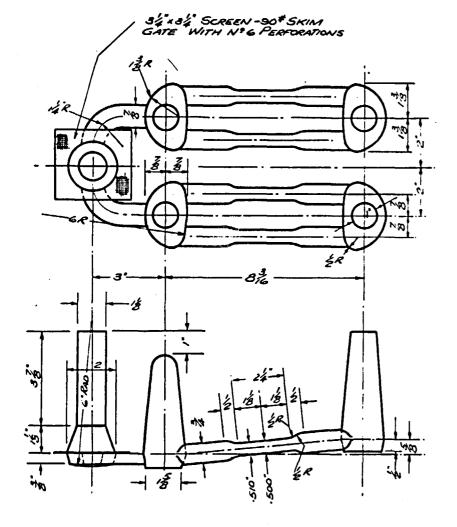
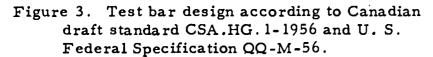




Figure 1.

1





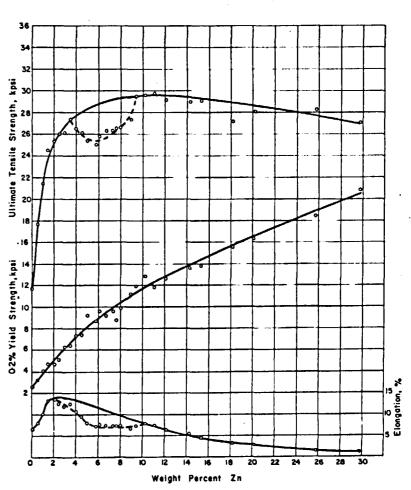


Figure 4.

Tensile Properties of Sand Cast Mg-Zn Alloys As Cast Condition (F)

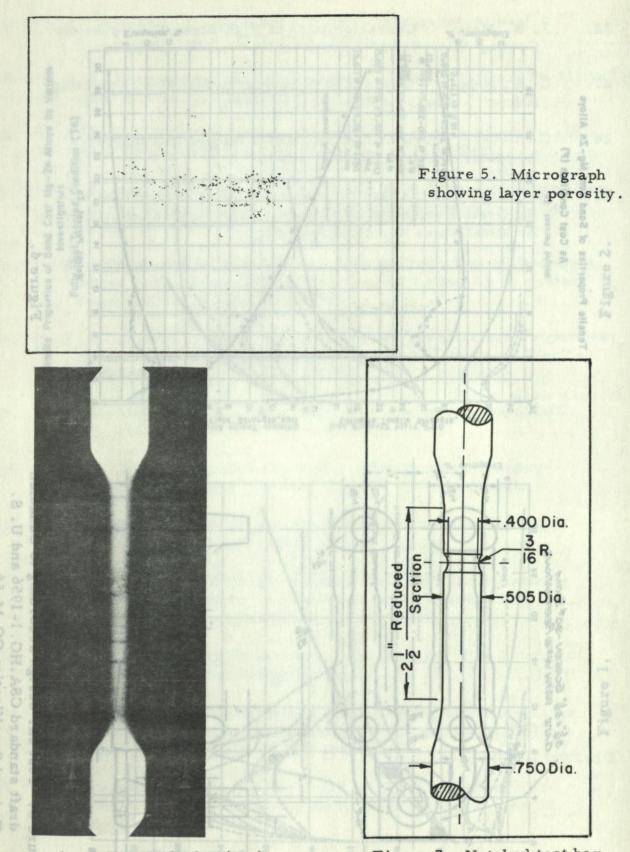
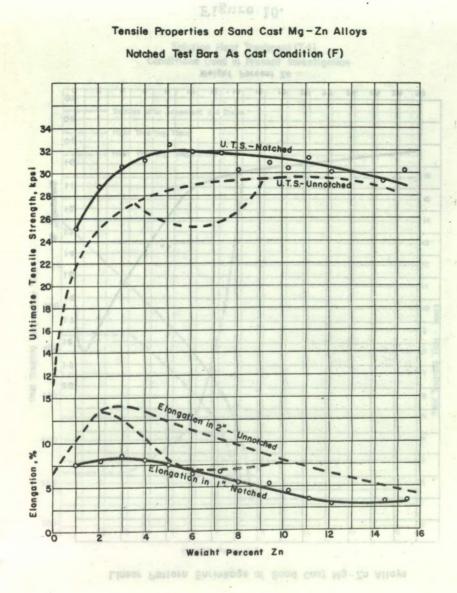
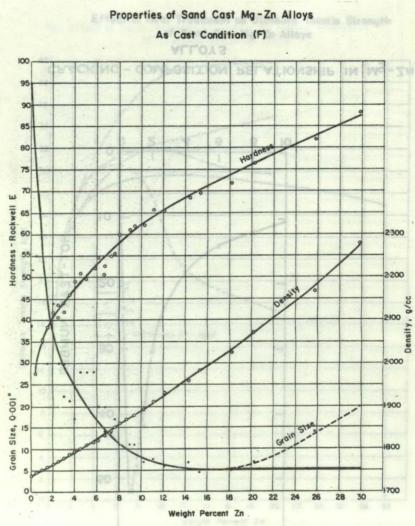


Figure 6. Radiograph showing layer porosity in gauge length of test bar. Figure 7. Notched test bar for tensile testing.

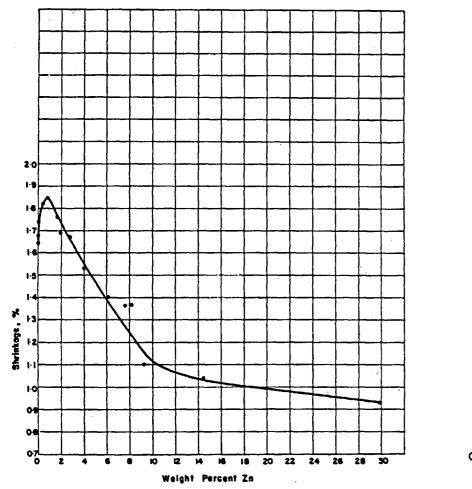


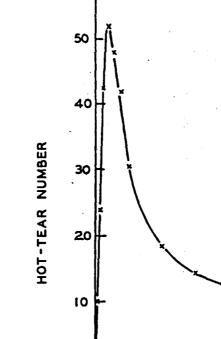


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Figure 8.





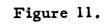


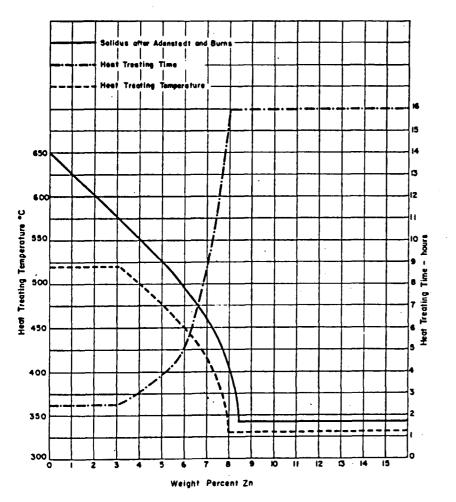
r1

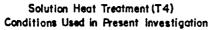
Linear Pattern Shrinkage of Sand Cast Mg-Zn Alloys

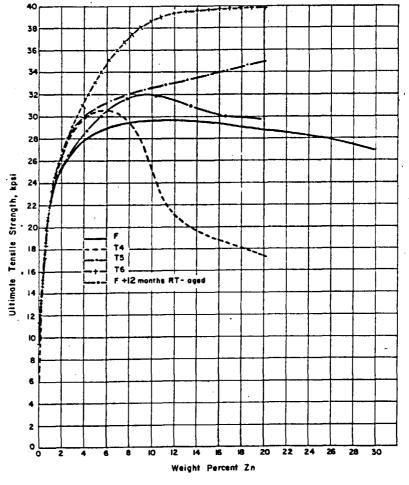
CRACKING - COMPOSITION RELATIONSHIP IN Mg-ZM ALLOYS

Figure 10.



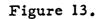


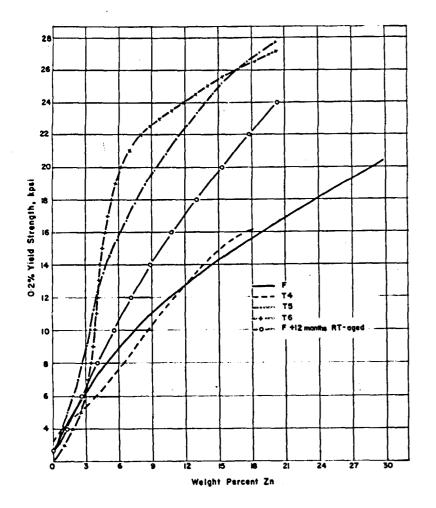




#### Effect of Heat Treatment on Ultimate Tensile Strength of Sand Cast Mg-Zn Alloys

Figure 12.





#### Effect of Heat Treatment on 0.2% Yield Strength of Sand Cast Mg-Zn Alloys

Effect of Heat Treatment on Elongation of Sand Cast Mg-Zn Allays

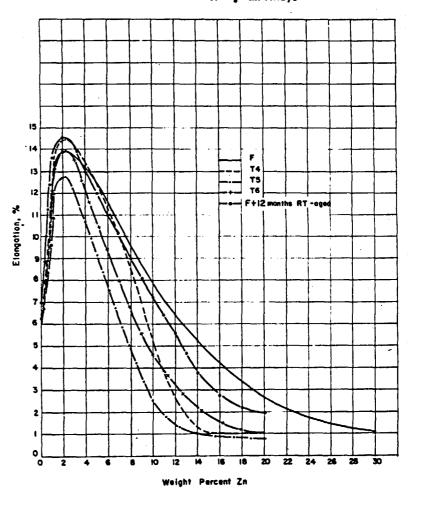
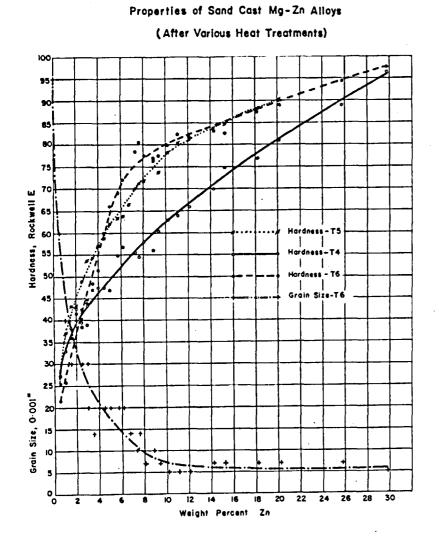
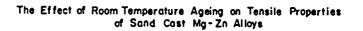


Figure 14.

Figure 15.

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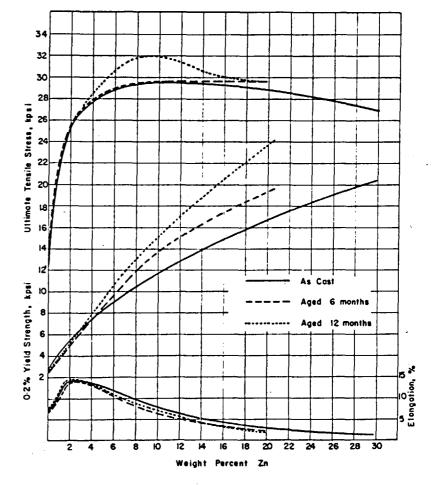


Figure 16.

Figure 17.

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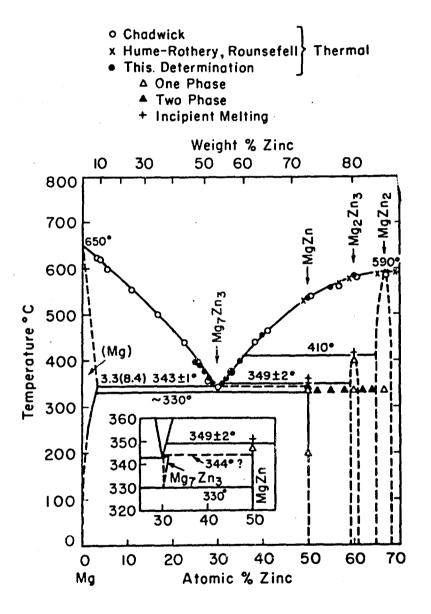


Figure 18. Partial phase diagram  $Mg-MgZn_2$  (according to Anderko et al<sup>(2)</sup>).

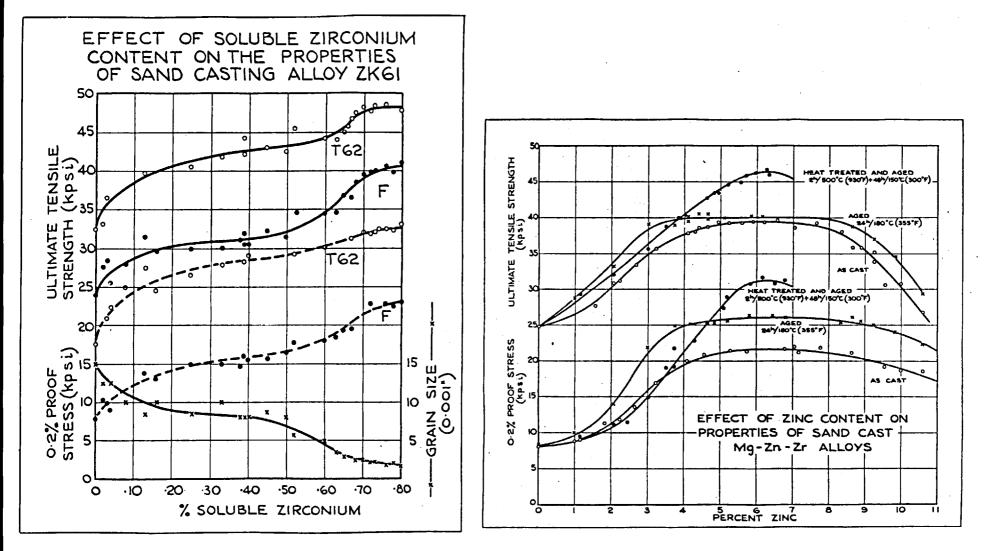


Figure 19. Effect of soluble zirconium content on properties of sand-casting alloy ZK61.

Figure 20. Effect of zinc content on tensile properties of sand-cast Mg-Zn-Zr alloys (containing over 0.7% soluble zirconium).

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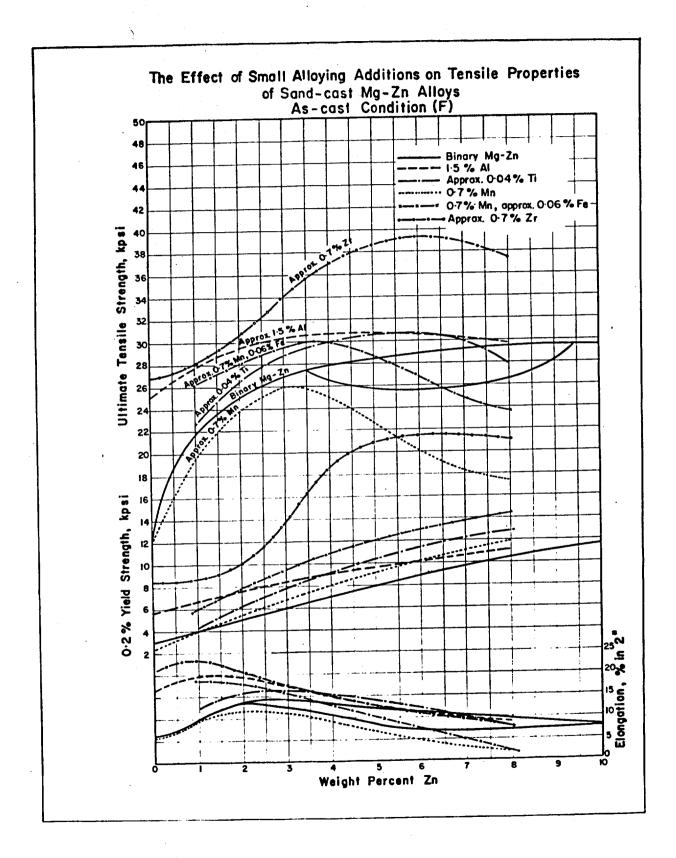


Figure 21.

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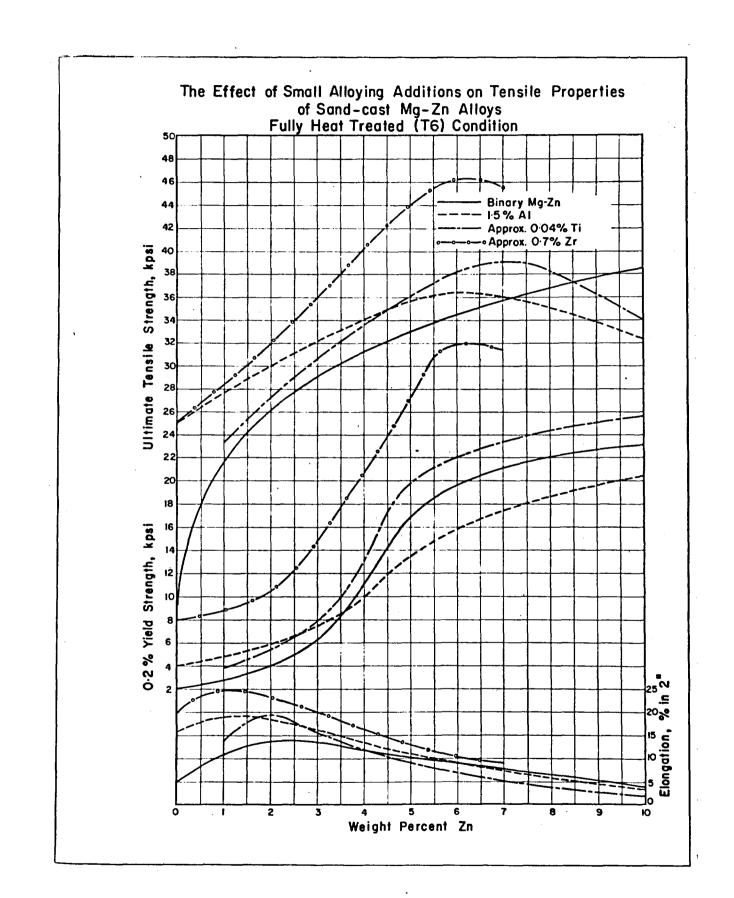


Figure 22.