FLUIDITY OF MAGNESIUM ALLOYS

by

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ABSTRACT

A vacuum fluidity apparatus was modified so that the fluidity of magnesium alloys could be satisfactorily determined.

The fluidity of magnesium-rich parts of alloy systems was determined. Those alloy systems investigated were:

1. Magnesium-aluminum binary system.
2. Magnesium-zinc binary system.
3. Magnesium-aluminum-zinc ternary system.
4. Magnesium-zinc-zirconium system.
5. Magnesium-thorium-zirconium system.
6. Magnesium-rare earth-zirconium system.
7. Magnesium-rare earth system.

Fluidity was determined as a function of temperature for each alloy. Then fluidity at 1400°F was plotted as a function of alloy content. Fluidity at 100°F superheat was plotted for those alloy systems for which the liquidus temperature had been established. Comparison of commercial alloys with the experimental alloys was good. Fluidity curves as a function of alloy content were found to vary as the inverse of the computed, non-equilibrium freezing range curves. Addition of zirconium to magnesium alloys lowered the fluidity at low alloy content, but slightly raised the fluidity at alloy content above four percent by weight.

Thesis Supervisor Howard F. Taylor, Professor of Metallurgy

Lind (Metall.) Jan. 30, 1957

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I. INTRODUCTION

One of the important factors concerning foundry technology is the fluidity of the alloy used. This research program has been directed at the study of the fluidity of magnesium alloys.

"Castability" is frequently considered to be synonymous with "fluidity", but actually castability embraces such factors as high fluidity and freedom from microporosity and hot tears. Mechanical properties are functions of alloy content and heat treatment, but good mechanical properties cannot be realized in poor castings; so castability is indirectly related to strength, ductility, and other mechanical properties.

Fluidity is defined[1] as the ability of a liquid metal to flow readily as measured by the length of a standard spiral casting. Fluidity refers to the property of a metal which allows it to flow freely and evenly into a mold and fill it before such freezing occurs as would offer an obstruction to its further flow.

The apparent fluidity characteristics of various alloys are well known to the foundryman. Some alloys are "easy" to cast; others extremely difficult. This difference appears to be fundamentally related to the mode of solidification of the alloy. Pure metals and some alloys solidify substantially at one temperature; they have definite melting points. Most alloys of commercial importance, however, freeze over a range of temperatures. The existence of this freezing range promotes dendritic or "mushy" solidification. In general, with increasing degree of "mushiness" the fluidity decreases.

Fluidity test results depend on mold and other variables as well as metal variables. These variables are important insofar as they affect mode of solidification, rate of freezing, type of mold filling, etc.

This study is an effort to extract the important factors affecting fluidity of magnesium alloys with the objective of greater understanding of these factors.

Fluidity studies reported herein have been directed at:

1. Obtaining a fluidity test for magnesium alloys which controls closely metal and mold variables.

2. Applying the fluidity test apparatus to compare the relative fluidity values of various magnesium alloys.

3. Determining the basic variables underlying the fluidity of magnesium alloys as a step to the understanding and improvement of their fluidity.

II. LITERATURE SURVEY

ANALYTICAL TREATMENTS

In fluidity studies by Kondic and Kozlowski[2] the variables associated with fluidity were listed in three classes: (1) metal variables (heat content, density, surface tension, viscosity, crystallization behavior, etc.), (2) mold characteristics (thermal properties, surface characteristics), and (3) conditions of the experiment (head of pressure, type of mold channel, etc.).

Fluidity calculations have been made by many investigators. The fluidity of a pure metal can be predicted by using a formula derived by Portevin and Bastien[3] (1932); however, the variables of solidification behavior make such predictions for alloys difficult at present. Ruff[4], in his calculations, used the laws of hydrodynamics after determining that fluid metal in a mold behaves like an ordinary incompressible fluid with turbulent flow characteristics. Ruff's assumptions and extrapolations in determining resistances to turbulent metal flow have been found to be faulty[5,6], thus affecting his calculations of fluidity. Klyachko and Kunin[7] (1949) calculated fluidity based on (1) factors affecting time metal is in liquid state and (2) factors affecting speed of molten metal. They studied fluidity variables including pressure head, superheat, speed of liquid metal, and hydraulic resistance to velocity. They concluded that superheat was the most important and influential variable.

Rightmire and Taylor[6] have corrected and expanded Ruff's theory relating fluidity of molten steel to the laws of turbulent flow hydrodynamics. The authors indicate that a preponderous amount of fluidity data supports this newer theory. The theory shows that flow ceases before the metal has lost appreciable latent heat of fusion or flow ceases just below the liquidus. The theory indicates that the temperature of pouring (or superheat) is the most important independent variable in fluidity measurement, and that other effects are relatively small.

In 1953 Ragone[8] made a detailed analytical study of fluidity of metals. His apparatus for experimentally checking his calculations consisted of a new type of fluidity tester wherein the liquid metal was drawn into a tube by a constant partial vacuum, thus controlling the head of liquid metal over the mold. Ragone concluded that in tubes of small diameter (i.e. main resistance to heat flow at the metal-mold interface) the fluidity of a pure metal at its melting point varied linearly with tube diameter; but in tubes of larger diameter or in insulating molds the fluidity varied as the nth power of the tube diameter where n approached 2 as a limit. In pure metals a substantial part of the fluidity is attained after the metal has reached its melting point. For alloys the fluidity was found to vary inversely as the solidification range (liquidus to solidus); and when the solidification range was large, the major portion of the fluidity was due to superheat above the liquidus.

FACTORS AFFECTING FLUIDITY

The amount of superheat (heat content) generally has the major effect upon fluidity for a particular alloy.[2,6,8,9,10] Rightmire and Taylor[6] state that the effect of composition on fluidity is almost entirely due to the effect on the amount of superheat through change of solidification temperature with composition. For consistent results with any fluidity test the pouring temperature and the mold temperature (if the melt is poured into a hot mold) must be controlled accurately.[11] Unfortunately above 1400°F some burning and oxidation occur and misruns are more frequent when working with magnesium.

Surfact tension and surface films may affect fluidity in many ways: (1) surface tension may appreciably reduce the force pushing the molten metal,[6] (2) solid surface films increase the effective surface tension (in certain cases by a considerable factor),[14] (3) solid surface films may be torn off and cause mechanical interference to metal flow,[14] and (4) solid surface films may change heat transfer coefficients and affect the rate of cooling with a subsequent effect on fluidity.[6,14] Dr. W. C. Newell[15] in 1948 stated that ",.. surface film is the determining factor as far as fluidity is concerned." But more recent studies by Kondic[14] and Rightmire and Taylor[6] show that the effect of surface tension, though important, is small and that the effect of films may be greater, depending on the nature of the film and the shape and size of the cross section; but not nearly as important as temperature (superheat). In 1939 Eastwood and Kempf,[16] in their study of fluidity of aluminum alloys, determined that the design of the test mold was a critical variable, especially when fluidity measurements were used to predict castability of thin sections. They revised the accepted test mold so that the alloy was cast into a flat spiral

1/16 inch thick. This mold increased the Area/Volume factor by three as compared with previous test molds; thus placing more emphasis on the effects of surface tension and surface films. The results, though still somewhat erratic, coincided with foundry experience more closely than any other test at that time. Klyachko and Kunin[7] have stated that the fluidity test must conform to the type of casting anticipated, i.e. thinner sections increase the effects of surface tension and surface films.

Kondic and Kozlowski[2] investigated the effect of height of metal head above the fluidity mold. They found that when the height of liquid metal was more than three inches above the mold, variations in height had no effect on fluidity results. When the height of metal was below three inches, increasing the liquid metal head increased fluidity. They also found that fluidity of commercial pure aluminum in a cast iron mold was not critically affected by mold temperatures up to 300°C.

Numerous investigators have studied the influence of viscosity on fluidity. There is general agreement that for alloys solidifying at a constant temperature (eutectics, pure metals or compounds, etc.) fluidity varies inversely as the viscosity.[2] Of course, viscosity varies inversely with temperature, so this relationship bears out the importance of superheat. In alloys, however, when solidification takes place over a range of temperatures, the fluidity is often drastically reduced.[2,9,10] Portevin and Bastien[17] went as far as to say that this phenomenon was a rule of fluidity. However, numerous exceptions to this "rule" have been observed and many theories have been advanced to explain this variation of fluidity. Portevin and Bastien[17] stated that fluidity depended on the form of crystallization of alloys and that dendritic crystals decrease fluidity much more than crystals with convex faces. This was attributed to mechanical interference to liquid flow through the dendritic crystals. Another theory[14] proposed that changed in composition changed the viscosity of the liquid metal and thus affected fluidity. Knodic and Kozlowski[2] and Kondic[14] later (1950) stated that the effect of viscosity was of minor importance and that mechanical interference by dendritic crystals played a secondary role in influencing fluidity. They proposed that the main influence was the change of the properties (heat of fusion, melting point, surface tension, etc.) of liquid metal with changing composition. Ragone[8] and Kunin[18] agree that viscosity is a minor influence; however, Kunin states that viscosity and surface tension are manifest especially in small cross sections. As stated above in the paragraph on superheat, recent thought is that the effect of composition on melting point, and hence on superheat, is the major influence on fluidity of alloys.

H. J. Fisher[19] studied viscosity of molten mercury, the lead-tin system, and the antimony-cadmium system. Experimental results showed the applicability of Ewell and Eyring's relation, log viscosity varies inversely with temperature, to the lead-tin alloy system in the liquid state. Kinematic viscosity was determined for the lead-tin and antimony-cadmium systems at constant temperature and at constant superheat. Upon superimposing a velocity of flow versus composition curve on the curve of fluidity reported by Portevin and Bastien, Fisher[20] found appreciable differences. He attributed the differences to experimental conditions in that the velocity of flow curves were obtained under isothermal conditions and fluidity curves under non-isothermal conditions. The non-isothermal conditions imposed new variables especially (1) solidification range, (2) latent heat of fusion, and (3) liquid viscosity changes. It is interesting to compare viscosity from Fisher's work[19] with fluidity from Ragone's work.[8] This is shown in Figure 1. It appears that viscosity has some influence on fluidity, but there are other variables of more importance.

Fisher's experiments[19] showed that viscosity varied continuously above the liquidus, thus disproving the results of other investigators who had found a sharp rise in viscosity just above the liquidus.

Investigators[21] at Dow Chemical Co. have computed viscosities of liquid magnesium by applying Andrade's formula to find the viscosity at the melting point; and then, by applying Ewell and Eyring's relation, calculating values of viscosity for temperatures above the melting point.

FLUIDITY OF MAGNESIUM ALLOYS

The only published work on fluidity of magnesium alloys is that by Busk and Marande.[22] They studied strength, elongation, porosity, fluidity, and other properties in the magnesium-rich corner of the magnesium-aluminum-zinc ternary diagram. Their

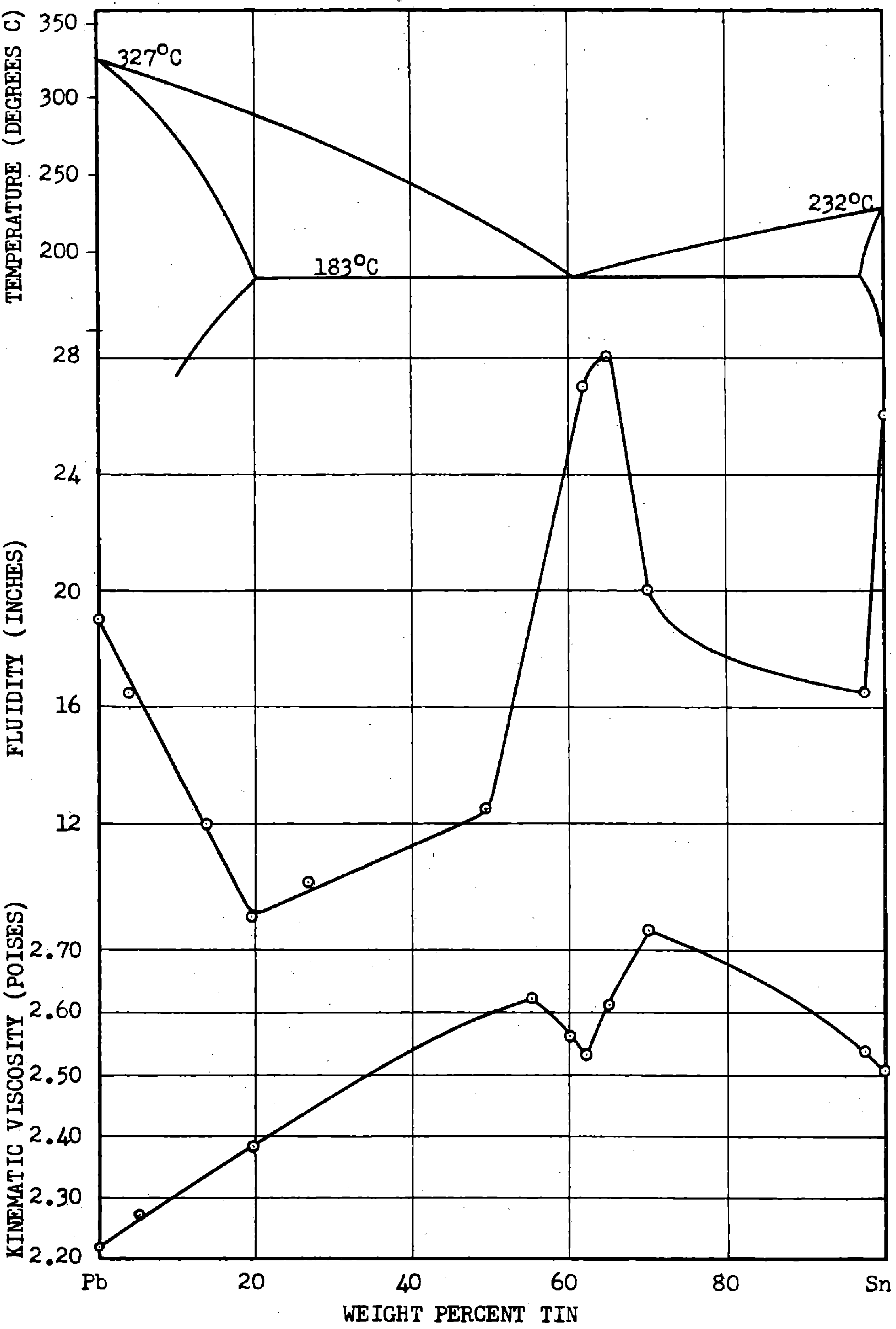

Figure 1 - Comparison of Fluidity and Kinematic Viscosity of the Lead-Tin System at Liquidus Temperature plus 50 degrees Centigrade.

fluidity diagram is shown in Figure 2.

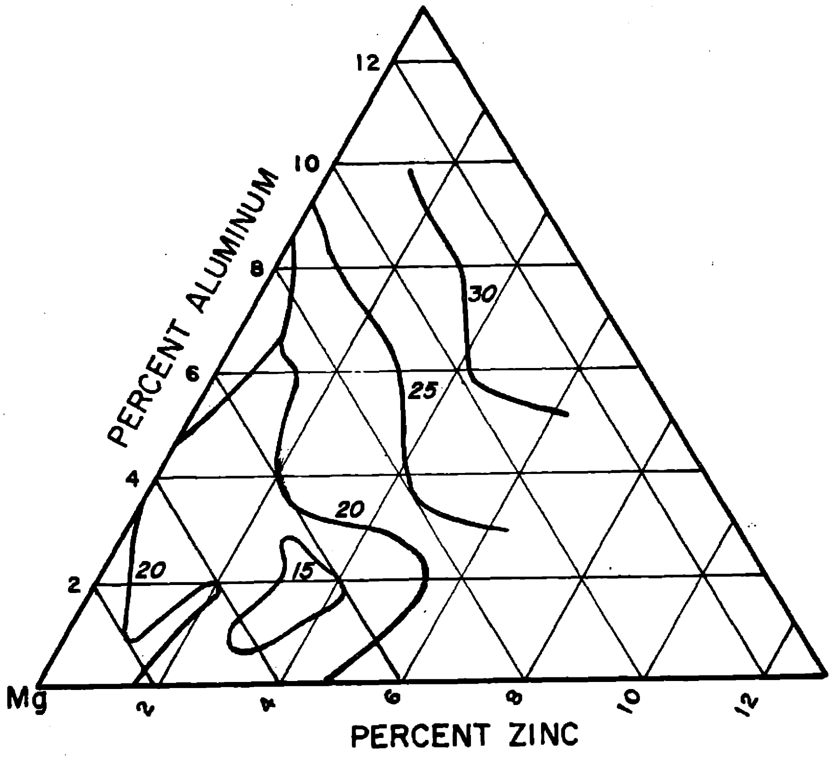

Figure 2 - Fluidity, inches. Length of Spiral as Obtained in Conventional Spiral Type of Fluidity Mold. All Alloys Poured at 1400°F.

GENERAL

Some isolated comments related to the fluidity of magnesium alloys have been found in this survey. These are listed below.

(1) Gas content in magnesium can consist of hydrogen, water, nitrogen, natural gas (furnace gas), oxygen or carbon dioxide; the most likely to reduce fluidity is hydrogen which is acquired by reaction of molten magnesium with water.[23] Juroff[24] and Busk and Marande[23] state that the best degassing method is to use dry chlorine gas and that this method gives consistently improved fluidity.

(2) Investigations[25] of the effect of various refining methods on the quality (homogeneity, lack of inclusions) of magnesium and certain magnesium alloys has shown:

(a) There is no difference in metal quality between using flux 230 or flux 310 as a refining agent.

(b) Chlorine is more effective than flux refining for increasing quality of metal.

(c) A high manganese content (above 0.6%) increased dross and lowered flowability through a screen.

(3) Magnesium-rare earth-zirconium molten alloys tend to oxidize in air more rapidly than other commercial magnesium casting alloys, resulting in pitting in cope surfaces or gross misruns if the melt is not properly protected by (1) adequate surface additions in the crucible (Dow No. 181 flux), (2) proper casting design to reduce turbulence, and (3) sufficient screening to remove oxide skins. Difficulties in casting magnesium-rare earth alloys are attributed to coarse grains and large amount of embrittling second phase material. Grain size is refined by addition of small amount of zirconium and elimination of manganese impurities. The amount of second phase material is reduced by reducing rare earth content from 6% to 3%. The resulting alloys are EK30 and EK31 (Magnesium, 2-4% rare earths, 0.55% zirconium). Addition of 3% zinc to these alloys does not increase castability.[26]

(4) In a review of die casting in Germany in 1950 it was found that castability of magnesium-aluminum-zinc alloys was enhanced with increased aluminum content. AZ91 alloy (Magnesium, 9% aluminum, 0.7% zinc, 0.2% manganese) was declared best for intricate thin-wall

casting. Decreasing aluminum content and increasing zinc content was found to impair castability.[27]

(5) Alloy EM61B (Magnesium, 6% rare earths, 0.8% manganese, 0.2% nickel, 0.02% tungsten) has very poor castability properties as compared with EK30A, EK30B, EK31A, and EZ33A, which have about the same castability properties.[28]

(6) Precipitate of zirconium in magnesium alloys can be caused by aluminum, silicon, or hydrogen and results in poor grain size, lowered mechanical properties, cradking and microporosity. "ZRE.1 alloy (Magnesium, 2.5% zinc, 2.5-3% rare earths, 0.6% zirconium) is by far the easiest to handle of all the magnesium alloys."[29] The temperature control of rare earth-zirconium-magnesium base alloys is important as more zirconium is lost if the molten alloy is held below 1350°F. A very small amount of aluminum will cause drastic loss of zirconium in these alloys.[30]

(7) Solid solubility of thorium in magnesium is estimated at 10% thorium. Eutectic phase of the magnesium-thorium phase diagram contained about 35% thorium and consisted of alpha phase solid solution of thorium in magnesium and theta phase containing about 55% thorium. Difficulties in dissolving zirconium in magnesium-zinc melts were improved by using graphite crucibles instead of ferritic stainless steel, zirconia, or magnesia crucibles.[31]

(8) In 1954 Burkett stated that berylium (0.0007% weight content) in die casting alloys apparently had no effect on castability, but it was a good oxidation suppressor supplementing the use of fluxes and sulfur dioxide. His theory was that a tight

elastic surface film of berylium oxide prevented access of air and hindered vaporization of magnesium.

III. PLAN OF RESEARCH

The purpose of this investigation is to 1) obtain a fluidity test for magnesium alloys which controls closely metal and mold variables, 2) apply this fluidity test apparatus to compare the relative fluidity values of various magnesium alloys, and 3) determine some of the basic metal variables underlying the fluidity of magnesium alloys as a step toward the understanding and improvement of their fluidity.

To accomplish these aims the vacuum fluidity tester developed by Ragone[8] will be suitably adapted for use with magnesium alloys.

Vacuum fluidity tests on various magnesium alloys will be made, and the fluidity values compared at constant temperature and at constant superheat.

The relation of certain metal variables (solidification range, mode of solidification, etc.) to the determined fluidity values will be examined in an effort to evaluate the importance of these variables.

IV. EXPERIMENTAL PROCEDURE

APPARATUS

The vacuum fluidity apparatus used by Ragone[8] was used in this study. The apparatus was adapted somewhat for use with magnesium and its alloys. A schematic drawing is shown in Figure 3 and a photograph in Figure 4.

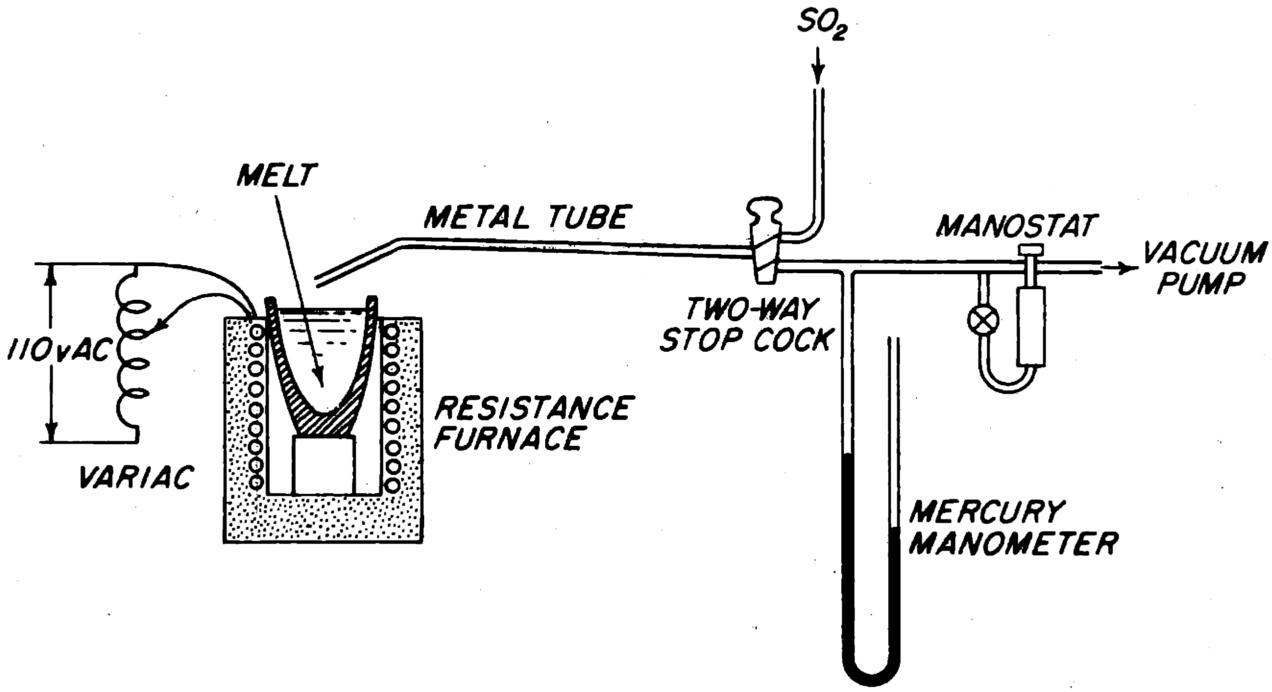

Figure 3 - Sketch of Fluidity Apparatus

The furnace is a resistance wire furnace. The voltage was controlled with a variac. Although this furnace was designed for lower melting alloys, it was found satisfactory for this work. Maximum temperature of molten metals attained with this furnace was about 1500°F; reaching higher temperatures was found to take a prohibitively long time.

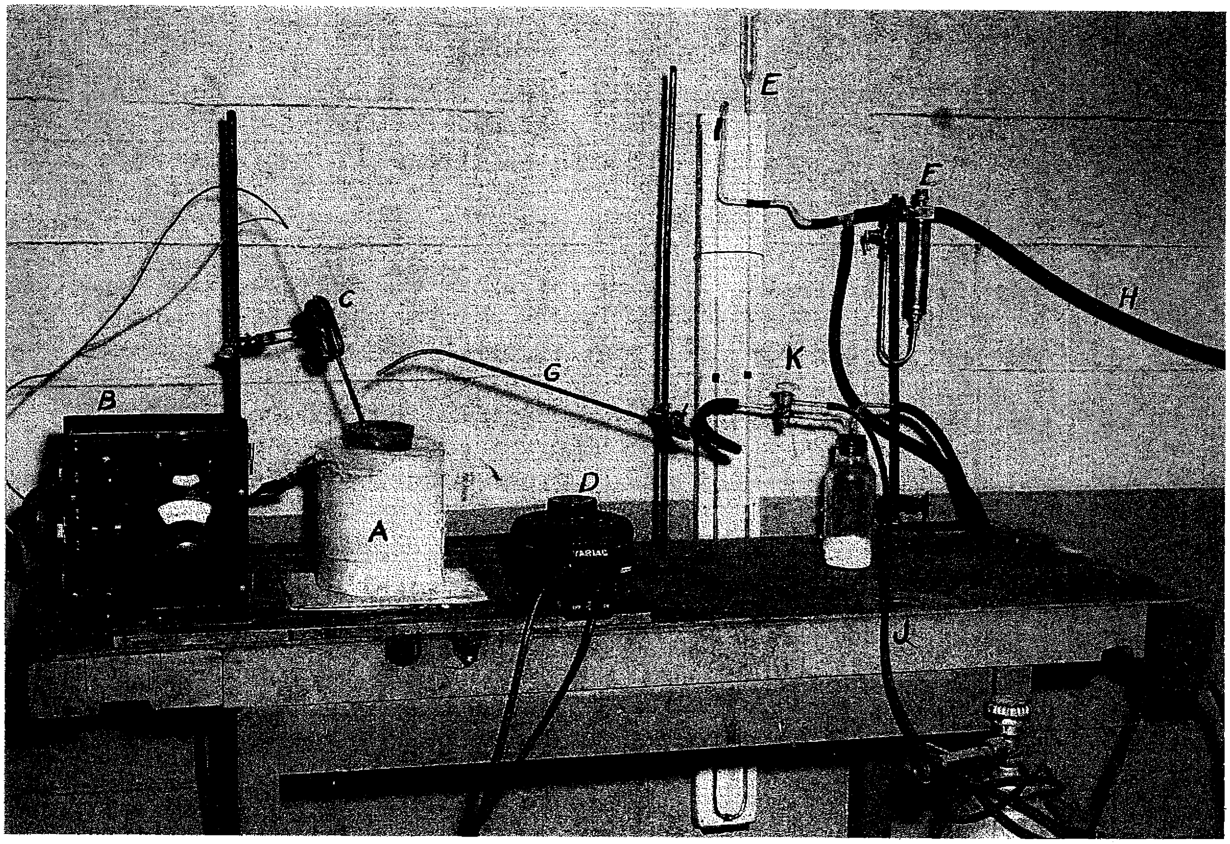

Figure 4 - Photograph of Fluidity Apparatus

A. Resistance-wire Furnace
B. Potentiometer
C. Thermocouple
D. Variac for Furnace Voltage
E. Manometer to Measure Vacuum
F. Manostat to Regulate Vacuum
G. Metal Tube
H. Rubber Hose from Vacuum Pump
J. Rubber Hose from SO_2 Bottle
K. Two-way Stop Cock

Choice of materials for tubing was limited by the reactivity of magnesium. Glass and vycor were tried with no success. Seamless stell tubing was used throughout this study because it did not react with magnesium and it could be obtained with satisfactory dimensional tolerances.

Tubing with an inside diameter of 0.126 - 0.002 inch was used. It was felt that larger bore size would decrease the surface area to volume ratio such that this study might not be applicable to thin-walled castings. Standard 1/4 inch, 16 gauge tubing is nominally 0.128 inch inside diameter, but the tolerances allow a variation of 0.020 inch in inside diameter. Figure 5 illustrates the variation in fluidity when the diameter is increased from 0.126 to 0.142 inch. In order to minimize this variable, each lot of

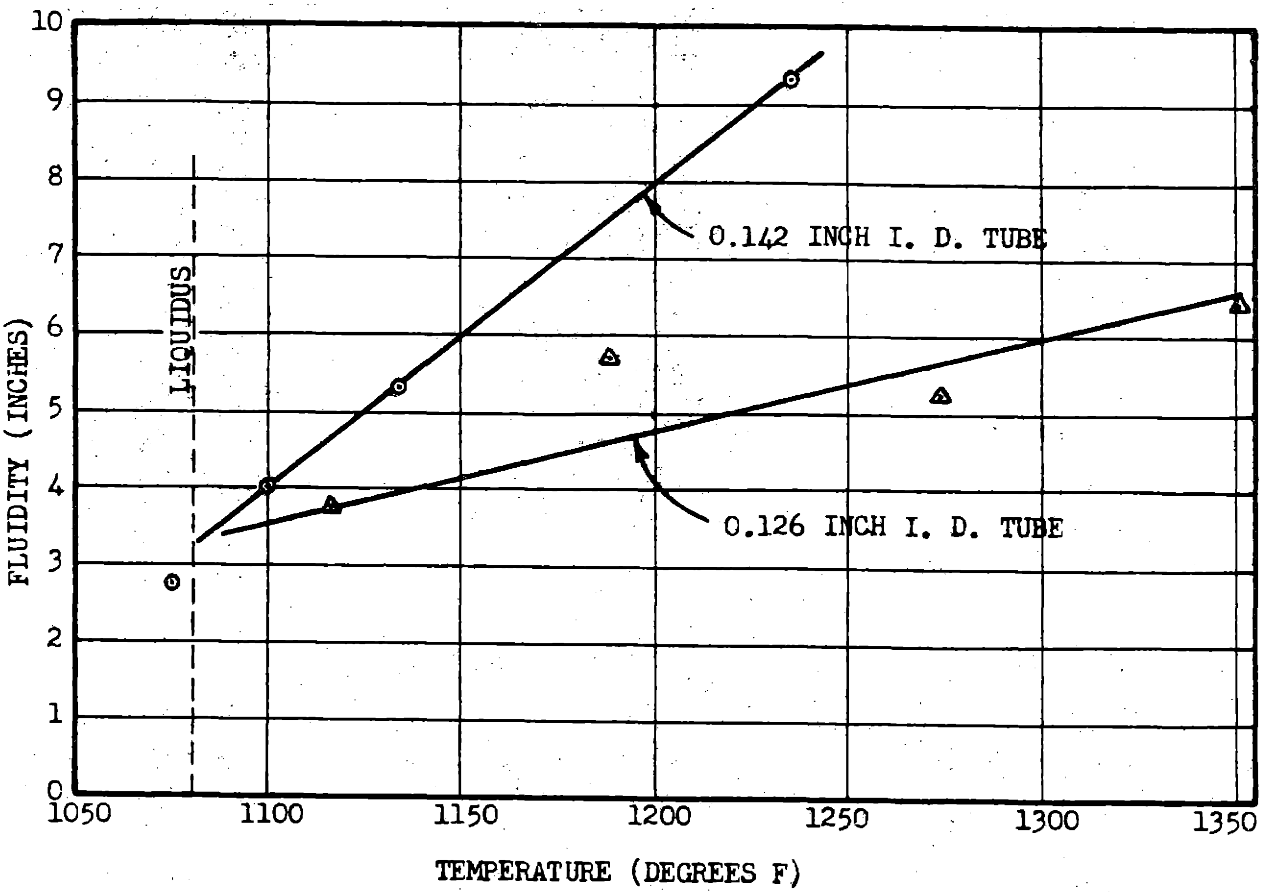

Figure 5 - Fluidity versus Temperature Curves for Magnesium, 13% Zinc, 3.5% Aluminum Alloy in Steel Tubes of Different Size. Liquidus Temperature Determined from Cooling Curve.

1/4 inch, 16 gauge tubing was inspected prior to purchase to insure that the inside diameter was satisfactory for this study.

Temperature measurements were made with a chromel-alumel thermocouple and a potentiometer. The thermocouple was used without a shield except when zirconium was alloyed in the magnesium melt. In order to prevent possible precipitation of zirconium by the alundum thermocouple sheath, a plain carbon steel tube, closed at one end, was used as a shield.

Degassing of the molten melts with chlorine was accomplished by allowing chlorine gas to pass from a cylinder through a rubber hose, then through a stainless steel tube to a graphite tip about six inches long which was immersed in the melt. Flow of chlorine was regulated by a valve next to the cylinder stop valve. A steel tip was tried on the degassing apparatus, but the molten metal would freeze in the hole even though the tip was heated to red heat. The subsequent build-up of chlorine pressure in the tube was dangerous as the frozen plug could remelt and cause a violent rush of chlorine into the melt. A heated graphite tip prevented this situation. The graphite tip was machined from 1/2 inch diameter graphite electrodes.

PROCEDURE

Briefly, liquid metal was drawn from the crucible into a tube by a vacuum. The metal flow stopped when any section of the flowing stream was sufficiently solid to prevent the passage of more liquid. The length of solidified metal in the tube was measured; this length was taken as a measure of the fluidity. One run was made for each alloy. Each run consisted of six fluidity tubes taken at different temperatures as the melt cooled from 1500°F.

Preparing the Melt

Burning of the magnesium metal was prevented by using fluxes. Dow 310 flux was used for magnesium alloys containing aluminum, zinc, or rare earths, and Dow 220 flux was used for all alloys containing zirconium.

Alloying was generally accomplished shortly after meltdown; except zirconium which was alloyed at 1450°F. Total wieght of each melt was about three hundred grams.

Most alloying elements were of commercial purity except aluminum which was high purity aluminum. The thorium was obtained in the form of a master alloy which contained about twenty-seven percent thorium. Zirconium "hardener" containing about half magnesium and half zirconium was used for alloying zirconium. Mischmetal was the source for rare earths additions. Mischmetal consists of about fifty percent cerium, about twenty-five percent lanthanum, and remainder iron, silicon, and other rare earths.

Preliminary fluidity tests with aluminum alloy 195 (aluminum, 4.5 percent copper) showed that the fluidity of a pure alloy was lower than the same alloy with some gas (hydrogen). In order to eliminate this possible variable from the magnesium fluidity results, degassing was accomplished on all melts except those containing zirconium. Because of the fine grain structure of magnesium-zirconium alloys, it is generally felt that degassing is not necessary; and in this study degassing was not used with zirconium because of the adverse effects it might have on the solubility of zirconium. Degassing was accomplished by bubbling chlorine through the melt for a minimum of five minutes when the melt was at approximately 1350°F.

Preparing the Tubes

The steel tubing came with a coating of grease inside and outside. This grease was removed by soaking each tube in trichloroethylene for about fifteen minutes. The end of the tube was then bent so that it could enter the crucible and still keep the major portion of the tube horizontal. The tube was dried and kept dry until it was used.

The radius of the curve in the bend in the tube can affect the fluidity accuracy. Ragone[8] found that the initial velocity of metal flow, when using zinc under similar conditions as in this study, was about 250 centimeters per second. Calculations with pure magnesium show that a velocity of about five hundred centimeters per second is attained under the conditions used in this study. These speeds could produce a cavitation effect in the bend of the tube; and, if the cavitation were severe enough, the length of flowing metal would contain vacuum pockets causing erratic fluidity results. Reducing the velocity of the metal and increasing the radius of the bend help to eliminate cavitation. Control of velocity is discussed later. In this study, bend radii of about one inch were attempted, but the radius was increased progressively until a radius of about four inches produced satisfactory results. Cavitation was not eliminated entirely, but its effect was restricted to the area of the bend.

The tubes were dried with dry nitrogen (-24°F dew point) or by heating in a large core treating furnace to 350 to 400°F. It was felt that the furnace-dried tubes would cool fast enough so that tube temperature would not affect fluidity results. However, when the melt cooled rapidly and fluidity samples were taken rapidly, the tubes did not cool sufficiently and fluidity results were somewhat higher than expected. Generally the melt cooled rapidly at the beginning of each run when the crucible cover was removed and about the middle of each run when the furnace voltage was turned down. Some fluidity versus temperature curves had two shallow humps when furnace-dried tubes were used. In drawing these curves more weight was given to the lower fluidity values at the start, middle, and end of the run. When tubes dried with nitrogen were used, no such consistent deviation of fluidity curves was noted.

It was found that the tubes could be used again after the bent section was cut off and the magnesium alloy removed. There was no detectable difference in fluidity results when the tubes were used the second time. The tubes were too short to be used safely after they were used twice.

Fluidity Testing

After alloying was completed, the temperature of the melt was raised to about 1500°F. The vacuum pump was turned on and the manostat was adjusted to bring the vacuum to the proper value. This value was observed on the manometer. The first tube was installed in the apparatus and sulfur dioxide was allowed to pass through the two-way stop cock into the tube. The sulfur dioxide acted as an oxidation inhibitor in the tube. The temperature was allowed to drop. At about the proper melt temperature, the potentiometer was accurately read, the flux was parted on the surface of the melt, the tube was lowered into the melt, and the stop cock was turned to admit the vacuum to the tube. The tube was removed when metal ceased to flow, and the next tube was inserted in the apparatus for the next sample. Six tubes were used for each run; the tubes were used at about equal temperature intervals between 1500°F and the liquidus temperature, or between 1500°F and 1200°F if the liquidus temperature was not known. After the last tube was used, a sample for spectrographic analysis was poured. For some alloys the melt was heated again and a temperature versus time cooling curve was recorded. In some cases a good cooling curve showed deviations from which the liquidus and solidus temperatures could be approximated.

The effective head of metal (in inches of metal) is given by:

$$\Delta P_m = \Delta P_{Hg} \frac{\rho_{Hg}}{\rho_m} - \Delta Z$$

where: ΔP_m = effective head of metal measured in inches,

ΔP_{Hg} = difference in pressure between the inside of the tube and atmosphere measured in inches of mercury,

ρ_{Hg} = density of mercury,

ρ_m = density of metal,

ΔZ = difference in height between metal level in crucible and the level of the channel, measured in inches.

In these experiments ΔZ ranged from one half to one and one half inches. ρ_{Hg}/ρ_m for pure magnesium varied from 8.5 to 9.4 depending on the temperature. (Magnesium density determined by Dow Chemical Co.[33] was used.) The head of metal, ΔP_m, and also the initial velocity of the flowing metal, was controlled by adjusting the manostat, thus varying ΔP_{Hg}.

The effect of varying ΔP_{Hg} on the fluidity of pure magnesium at 1400°F is shown in Figure 6. At low pressures the change in fluidity with a change in pressure was large; but at ΔP_{Hg} values above ten centimeters, the change in fluidity was much smaller. However, if a high head of metal was used,

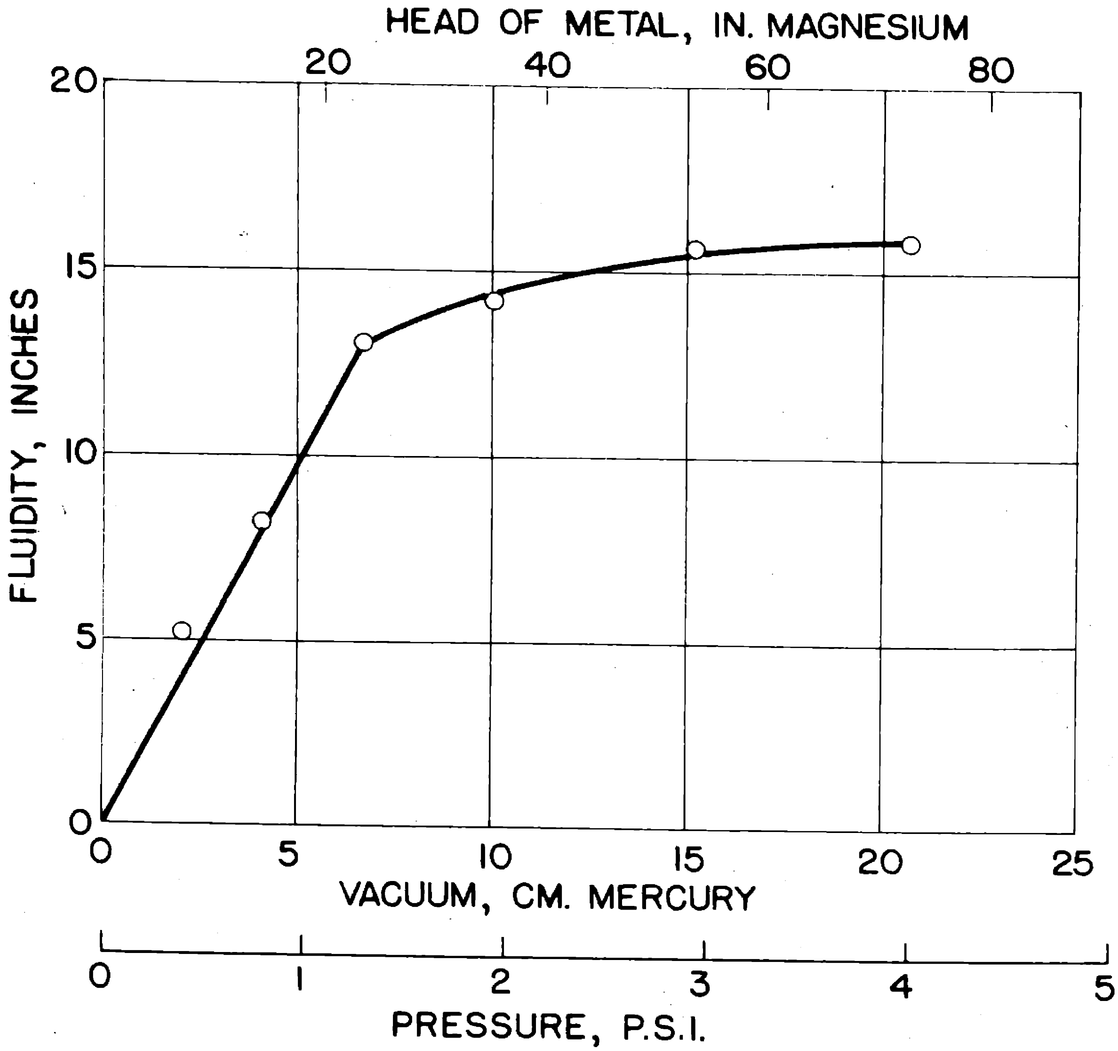

Figure 6 - Fluidity of Pure Magnesium at 1400°F as Pressure between the Atmosphere and the Inside of the Tube is Varied. All Abscissa Scales are Equivalent.

the velocity of the metal was high enough to produce excessive cavitation and make the results erratic; if a low head of metal was used, the metal might not flow far enough in these small tubes to give adequate length for comparison. It was found that a value of ΔP_{Hg} of fifteen centimeters or 5.9 inches was a satisfactory balance between the two extremes. ΔP_{Hg} was controlled to plus or minus 0.1 centimeter of mercury. This chosen value of fifteen centimeters of mercury results in a maximum variation of ΔP_m for pure magnesium of 48.3 to 55.4 inches of magnesium. The ΔP_m for magnesium alloys was slightly lower due to the increase in ρ_m, but the maximum variation in ΔP_m remained approximately the same. Figure 6 illustrates the fact that, in the range where ΔP_{Hg} is fifteen centimeters, the effect of variation of ΔP_{Hg} (or ΔP_m) on the fluidity is small. This is supported by the work of Kondic and Kozlowski.[2] Although the calculated head of metal varied with the density of the molten alloys, the pressure forcing the metal to flow remained constant for all tests of this study. This pressure was fifteen centimeters of mercury or 2.9 pounds per square inch.

At first a seal of scotch tape was used on the end of the metal tube. When the melt contacted the tape, it burned and allowed the vacuum to draw the metal into the tube. Examination of the metal in the tube showed a black skin inclusion which was the residue of the tape. This residue produced erratic fluidity results, and the use of a scotch tape seal was abandoned in favor of the stop cock manual control.

Analysis of Fluidity Tests

The length of metal in the tube was determined by measuring the total length of the tube and subtracting the void length in the tube. The void length was determined by inserting a thin rod into the tube and measuring the distance of the rod in the void space. The length of solidified metal could be measured to an accuracy of 1/16 inch.

The tubes were cut above the bend and the solidified metal was removed from the straight part of the tube. Figure 7 shows three typical tubes and six magnesium alloy samples extracted from similar tubes. Note that the length of these samples is

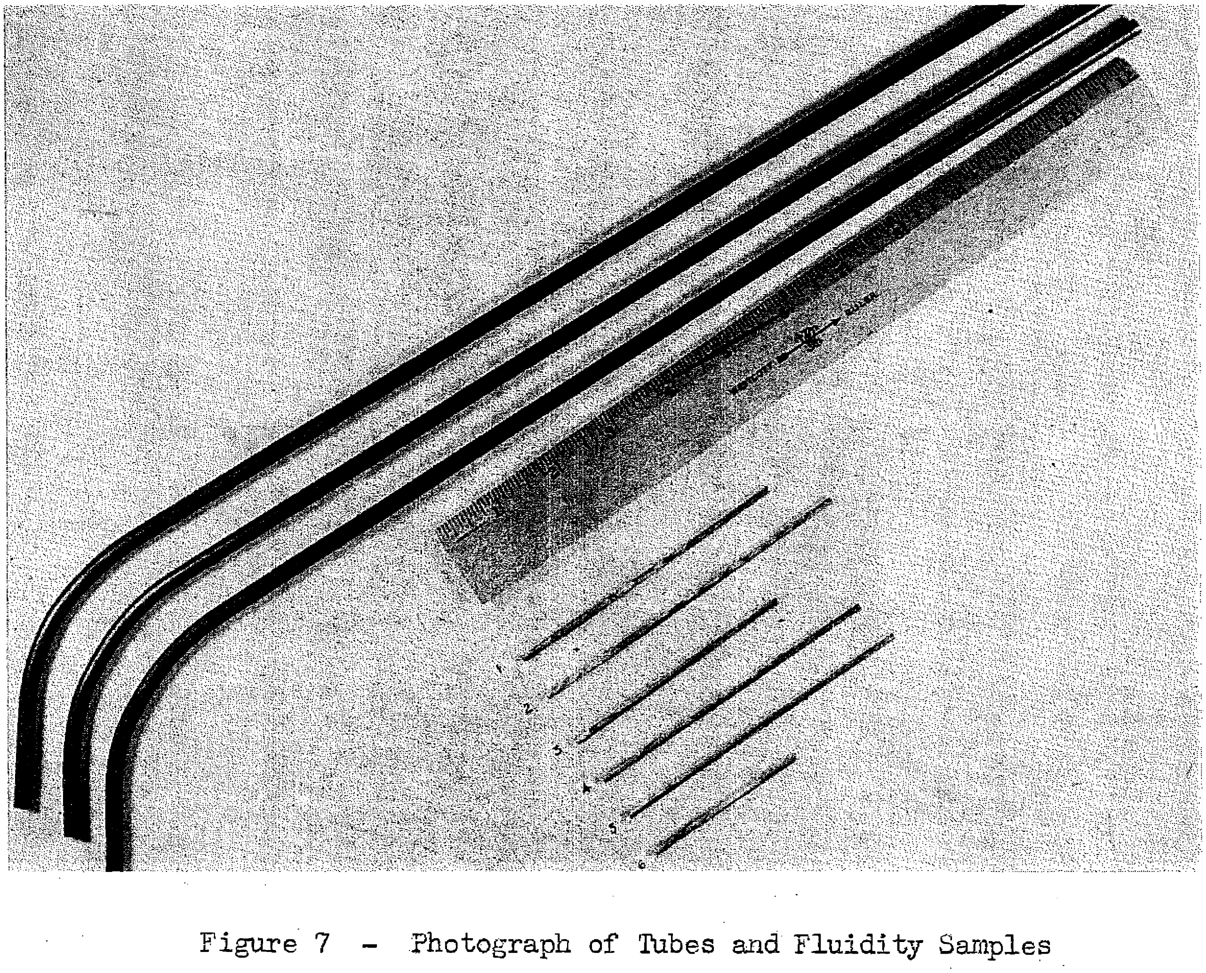

Figure 7 - Photograph of Tubes and Fluidity Samples

not indicative of the fluidity length, but depends on where the tube was cut. The diameters of all samples were measured with micrometers to insure that the diameter was within tolerances.

Spectrographic analyses were made on all melts by the Chicago Spectro Service Laboratory, Inc. They expect an accuracy of three percent of the amount of the element present. A few chemical analyses were made at M. I. T. for check purposes. These were in good agreement with the spectrographic results except for one zirconium analysis which was 0.09 percent higher by chemical method.

V. RESULTS AND DISCUSSION

Fluidity investigations were made for the following alloy systems:

(1) Magnesium-aluminum binary from 0 to 20 percent aluminum.

(2) Magnesium-zinc binary from 0 to 20 percent zinc.

(3) Magnesium-aluminum-zinc ternary from 0 to 20 percent aluminum-zinc.

(4) Magnesium-zinc-zirconium system from 0 to 20 percent zinc with 0.5 percent zirconium.

(5) Magnesium-thorium-zirconium system from 0 to 20 percent thorium with 0.5 percent zirconium.

(6) Magnesium-rare earth system from 0 to 20 percent rare earths.

(7) Magnesium-rare earth-zirconium system from 0 to 20 percent rare earths with 0.5 percent zirconium.

In addition, Dow Chemical Co. supplied commercial alloys for comparison with the results obtained from this study. Fluidity investigations were made for AZ92, ZK51, and ZH62 alloys.

Complete fluidity data are tabulated in Appendix A. The fluidity versus temperature curves for each alloy are plotted in Appendix B.

MAGNESIUM-ALUMINUM-ZINC SYSTEMS

The fluidity of the magnesium-aluminum system as a function of composition is shown in Figure 8, and the fluidity of the magnesium-zinc system as a function of composition is shown in Figure 9. The fluidity results of the magnesium-aluminum-zinc

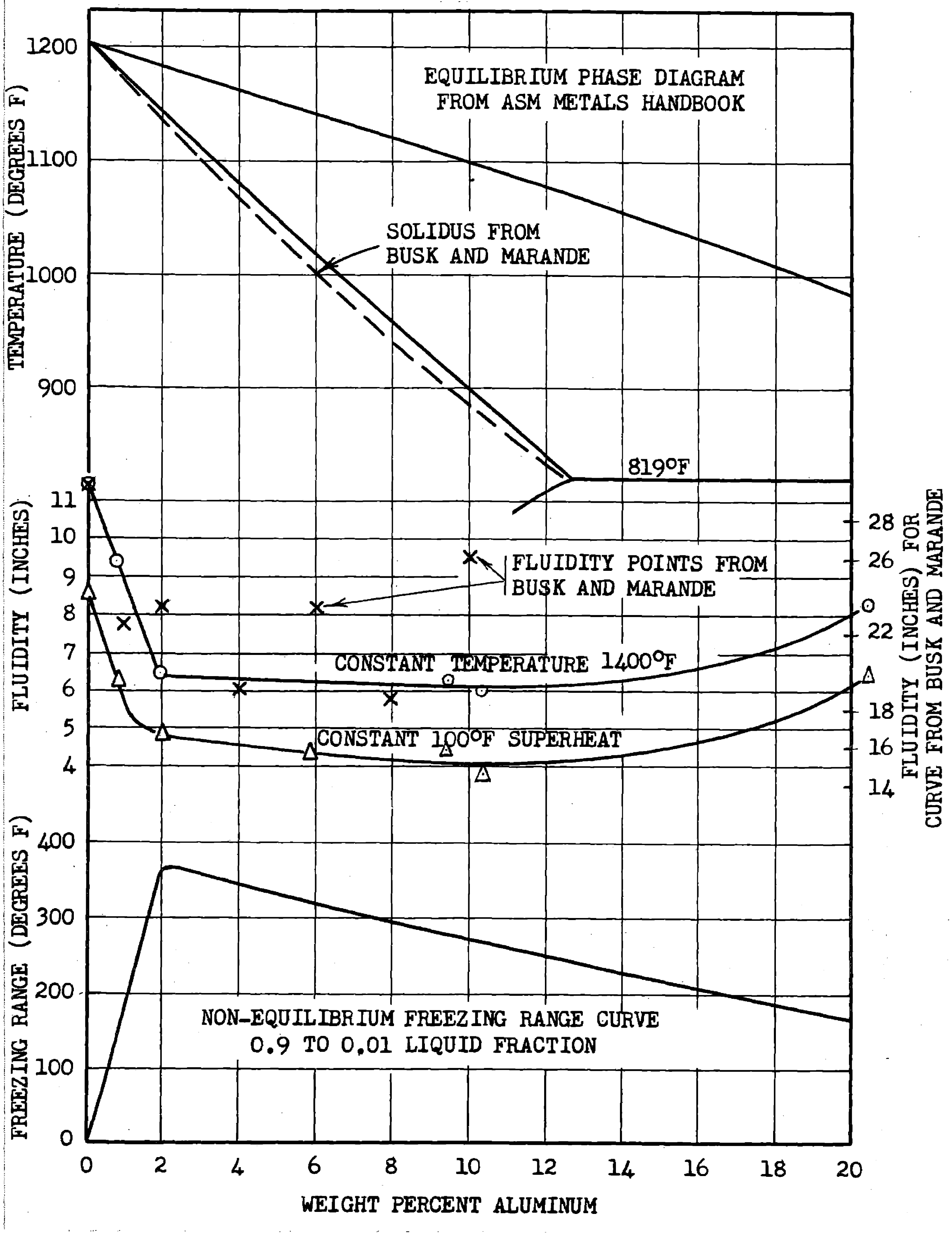

Figure 8 - Fluidity of Magnesium-Aluminum Binary System from 0 to 20% Aluminum.

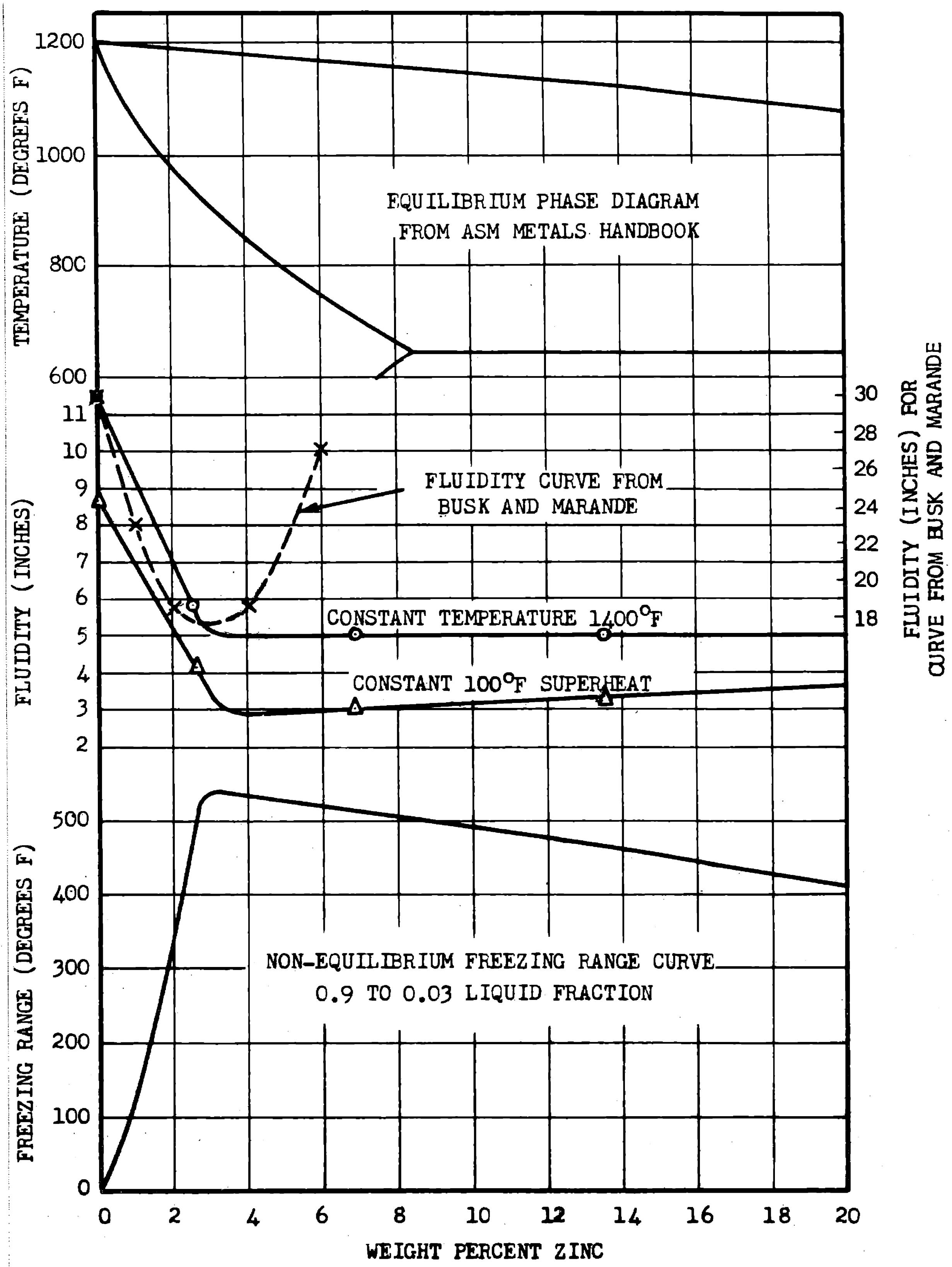

Figure 9 - Fluidity of Magnesium-Zinc Binary System from 0 to 20% Zinc.

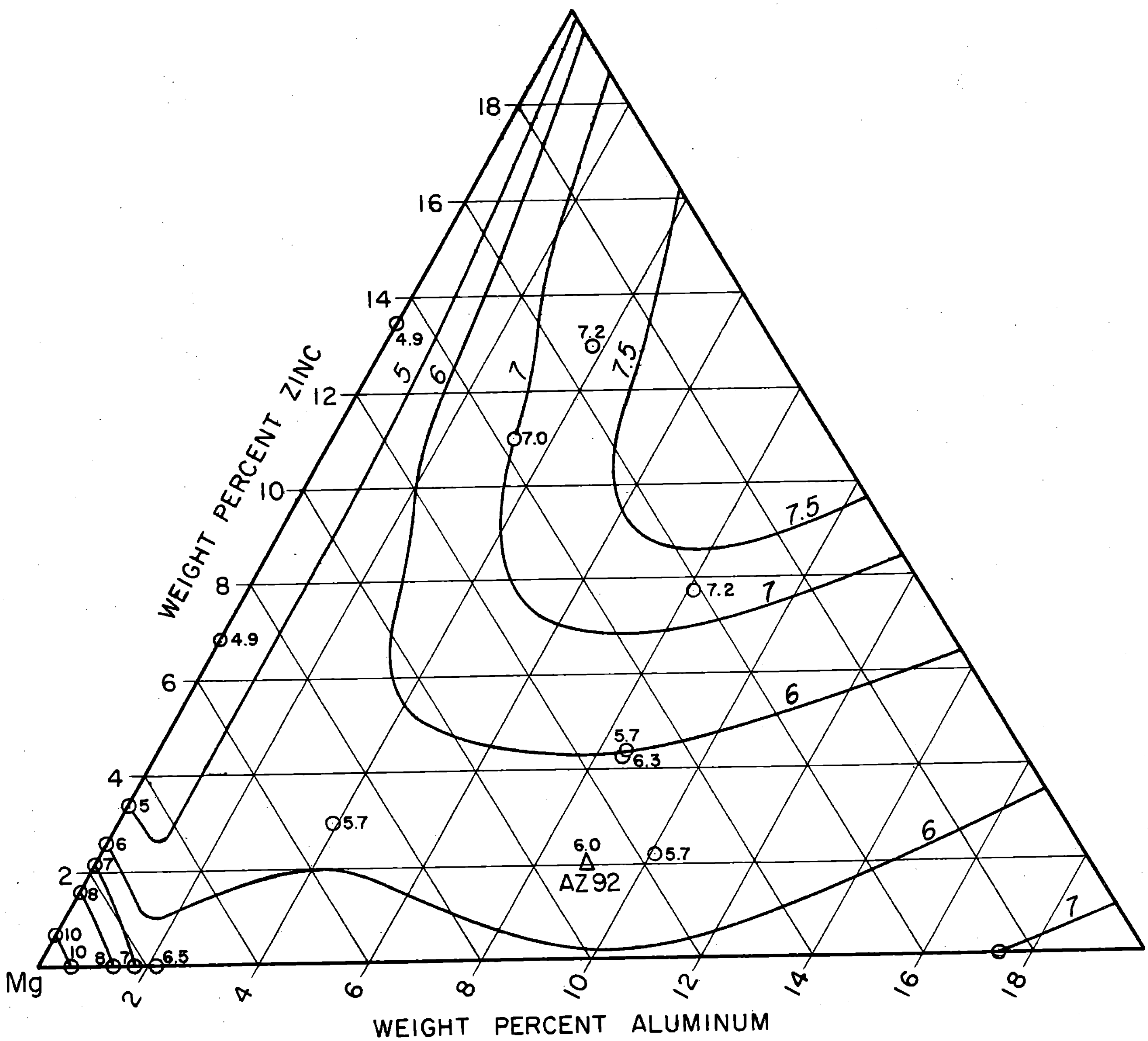

Figure 10 - Fluidity Curves at 1400°F for Magnesium-Rich Corner of the Magnesium-Aluminum-Zinc Ternary System. Fluidity in Inches.

ternary system are shown in Figure 10.

The fluidity data of Busk and Marande[22] are also shown in Figures 8 and 9. Busk and Marande used a standard spiral mold to determine fluidity. Their results should be different from those obtained with the vacuum fluidity apparatus due to: (1) the spiral mold is cast in sand, whereas a steel tube is used with the vacuum apparatus, (2) the section size of the spiral pattern is much larger than that used for the tests with the vacuum apparatus, and (3) the variables are much more difficult to control using the spiral mold.

The results plotted in Figures 8 and 9 agree with the predictions of the authors in the literature: the fluidity varied roughly as the inverse of the temperature range of solidification, i. e., the temperature difference between the liquidus and the solidus. Since the freezing of these alloys occurred rapidly, an equilibrium freezing was impossible; the temperature range of solidification was the temperature difference between the non-equilibrium liquidus and the non-equilibrium solidus. The non-equilibrium freezing range can be estimated for the eutectic type binary alloys using the equation:

$$\frac{W_L}{W_o} = \frac{X_o}{X_1} \frac{X_L}{X_L - X_S}$$

where: W_L = weight of liquid remaining

W_o = total weight of specimen

X_o = weight percentage of alloying element in original melt

X_1 = weight percentage of alloying element in remaining liquid

X_L = weight percentage of alloying element at eutectic composition

X_S = weight percentage of alloying element at point of maximum solubility.

This equation assumes (1) no diffusion in the solid state, (2) complete diffusion in the liquid state, (3) the equilibrium liquidus and solidus above the eutectic temperature are straight lines, and (4) there is no depression of the eutectic temperature. Naturally these assumptions are not valid, but work at M. I. T. indicates that the equation is a very good approximation. Realistic values for W_L/W_o to designate liquidus and solidus are chosen near one and zero. By assuming 0.9 for the fraction W_L/W_o, and then solving for X_1, the temperature of the remaining liquid could be found from the equilibrium phase diagram. This temperature corresponds roughly to the non-equilibrium liquidus temperature. Non-equilibrium solidus temperatures could be found by assigning small values to W_L/W_o, solving for X_1, and finding the corresponding temperature. Assuming W_L/W_o is 0.01 for the magnesium-aluminum non-equilibrium solidus and subtracting the solidus temperature from the liquidus temperature would give a non-equilibrium freezing range curve which is shown in Figure 8. The non-equilibrium freezing range curve for the magnesium-zinc system is shown in Figure 9 and was found by assuming that W_L/W_o is 0.03 at the non-equilibrium solidus.

Microscopic examination of etched rods of magnesium alloys after they were extracted from the metal tubes showed very clear evidence of coring. Every alloy examined showed signs of a second phase, probably eutectic, which increased in amount as the percentage of alloying element increased. A small amount of this second phase was found in the grain boundaries of the alloy analyzed at 0.74 percent aluminum, even though the maximum solubility of aluminum is 8.4 percent. This microscopic examination tends to emphasize the validity of the equation used above.

Although the fluidity curves for the magnesium-zinc binary system are fairly straight, the curves for the magnesium-aluminum system bend up at higher aluminum contents. This effect is caused by the proximity of the magnesium-aluminum eutectic at thirty-two percent aluminum; whereas the eutectic for the magnesium-zinc system is at about fifty-five percent zinc.

Using the non-equilibrium freezing range data for the binary alloys and the equilibrium freezing range (Figure 11) of the magnesium-aluminum-zinc ternary diagram derived from ASM Metals Handbook,[1] an estimated non-equilibrium freezing range diagram was constructed (Figure 12). The inverse of this diagram corresponds closely with the fluidity results for the magnesium-aluminum-zinc ternary shown in Figure 10. The rise of fluidity at compositions close to eight percent aluminum - eight percent zinc is caused by the effects of the ternary eutectic (at fifteen percent aluminum, thirty-six percent zinc) which begin to appear in this area as evidenced by the decrease in non-equilibrium freezing range in Figure 12.

It should be borne in mind that this particular modification of the vacuum fluidity apparatus involving steel tubes will result in a rapid chill of the freezing metal. The degree of chill will

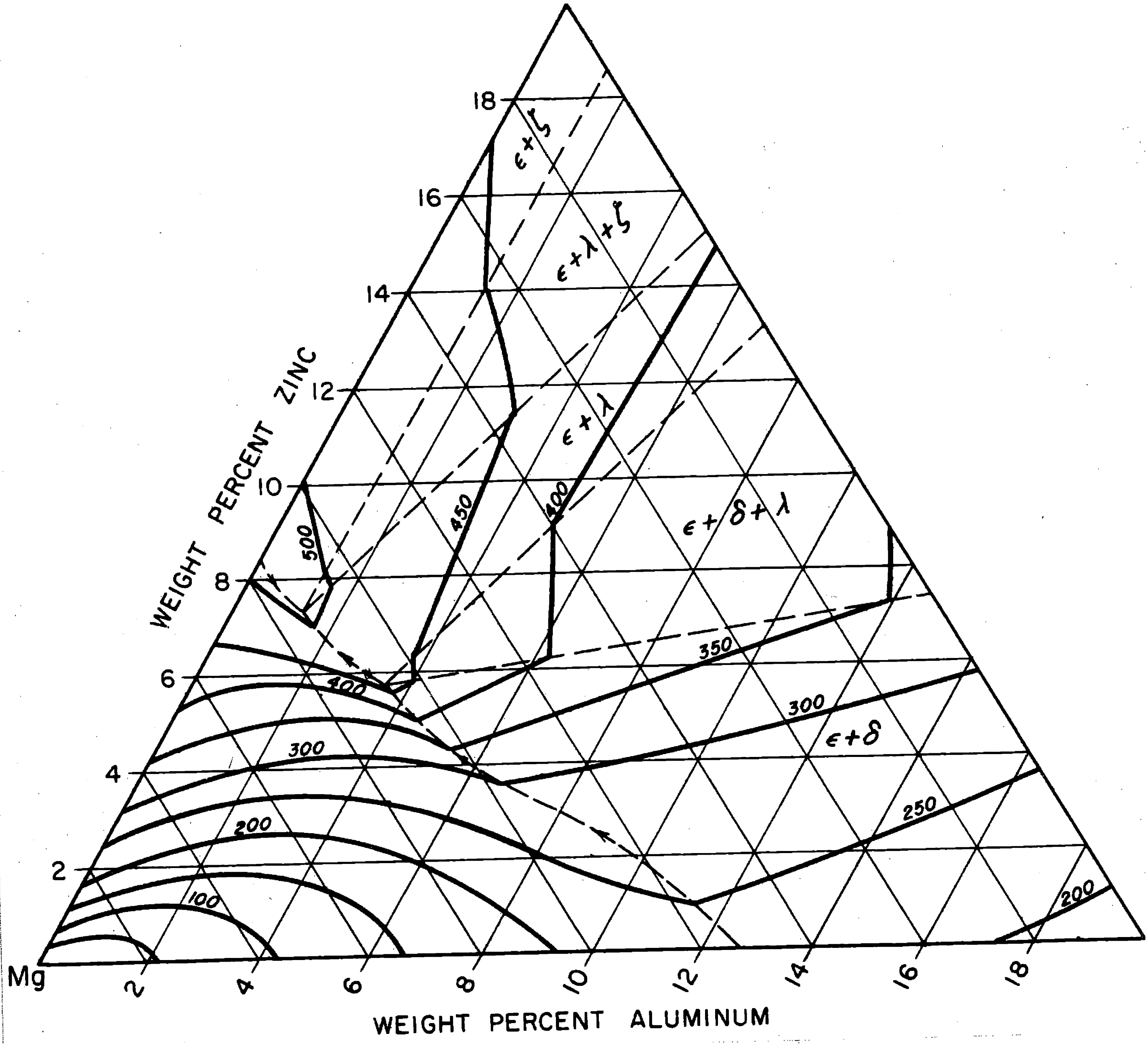

Figure 11 - Equilibrium Freezing Range (Degrees F) of Magnesium-Aluminum-Zinc Ternary Diagram in Magnesium-rich Corner. Taken from Liquidus and Solidus Surfaces in <u>ASM Metals Handbook</u>.[1]

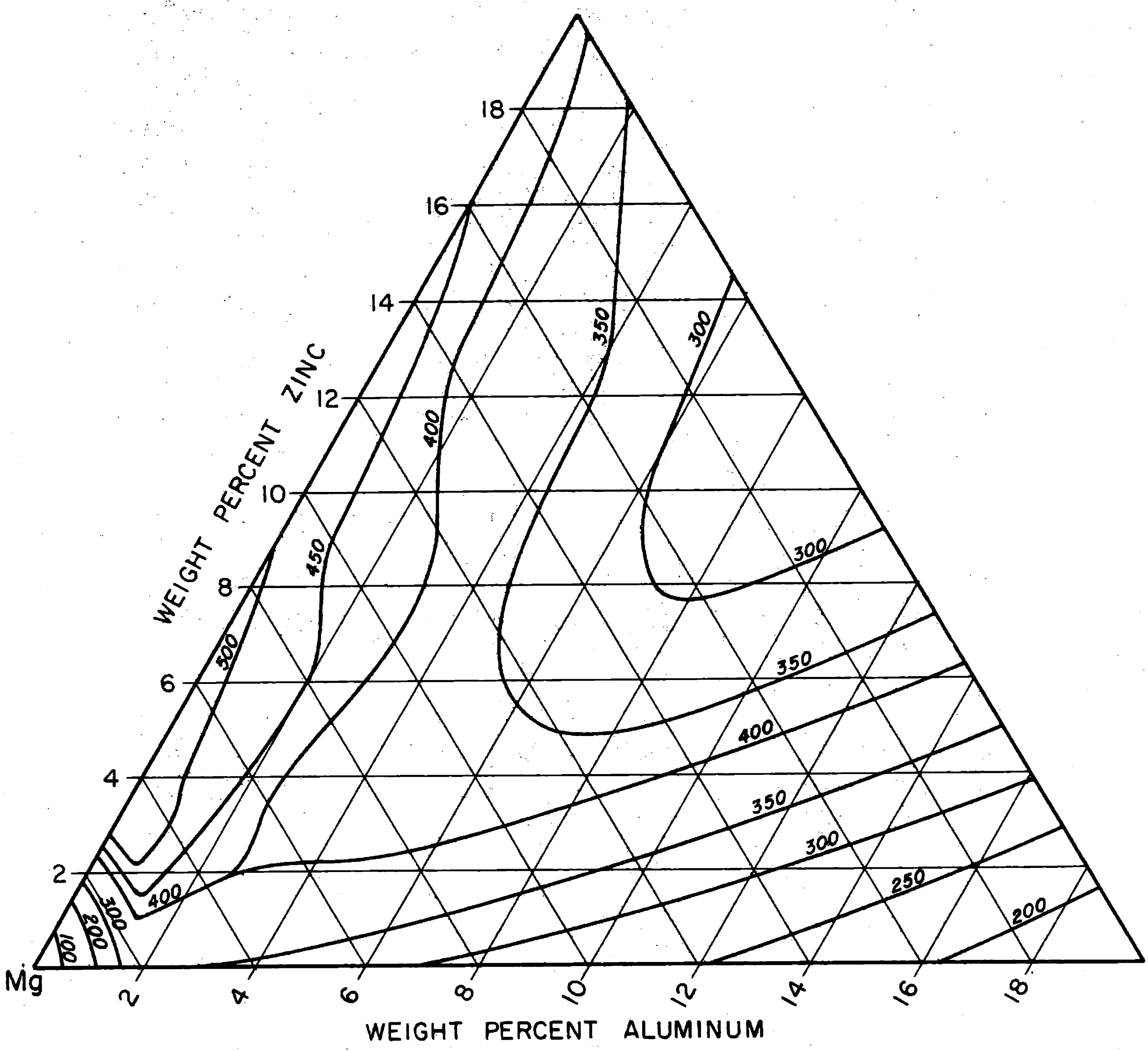

Figure 12 - Estimated Non-Equilibrium Freezing Range (Degrees F) for Magnesium-rich Corner of the Magnesium-Aluminum-Zinc Ternary Diagram.

determine the temperature range of solidification, and will affect the fluidity results. This fluidity variation may differ for various alloys. However, since the chill effect of this apparatus is similar to that experienced by the metal in a mold with thin cross section, the vacuum fluidity apparatus is considered better than the spiral mold when fluidity measurements are concerned with thin sections.

An inspection of Figure 5 and Appendix B will show that the effect of superheat on fluidity is not as great for small diameter tubes as it is for larger diameter tubes. This effect was shown experimentally by Ragone,[8] and was attributed primarily to the variation in the type of heat transfer resistance as the surface area-to-volume ratio was decreased. The similarity of the slopes of the curves in Appendix B indicates that the effect of superheat is approximately the same regardless of composition.

Commercial alloy AZ92 was tested. Its fluidity at 1400°F is shown in Figure 10. The fluidity value, 6.0 inches, is in good agreement with the results of this study. Part of the small difference may be caused by the higher manganese content of the experimental alloys (about 1.3 percent manganese as compared to 0.23 percent for AZ92).

MAGNESIUM-ZINC-ZIRCONIUM SYSTEM

The fluidity of the magnesium-zinc-zirconium system at 1400°F as a function of zinc content is shown in Figure 13.

The effect of zirconium on the magnesium-zinc phase diagram is not known, so the phase diagram of the magnesium-zinc binary system is shown instead. A curve at constant superheat cannot be drawn because

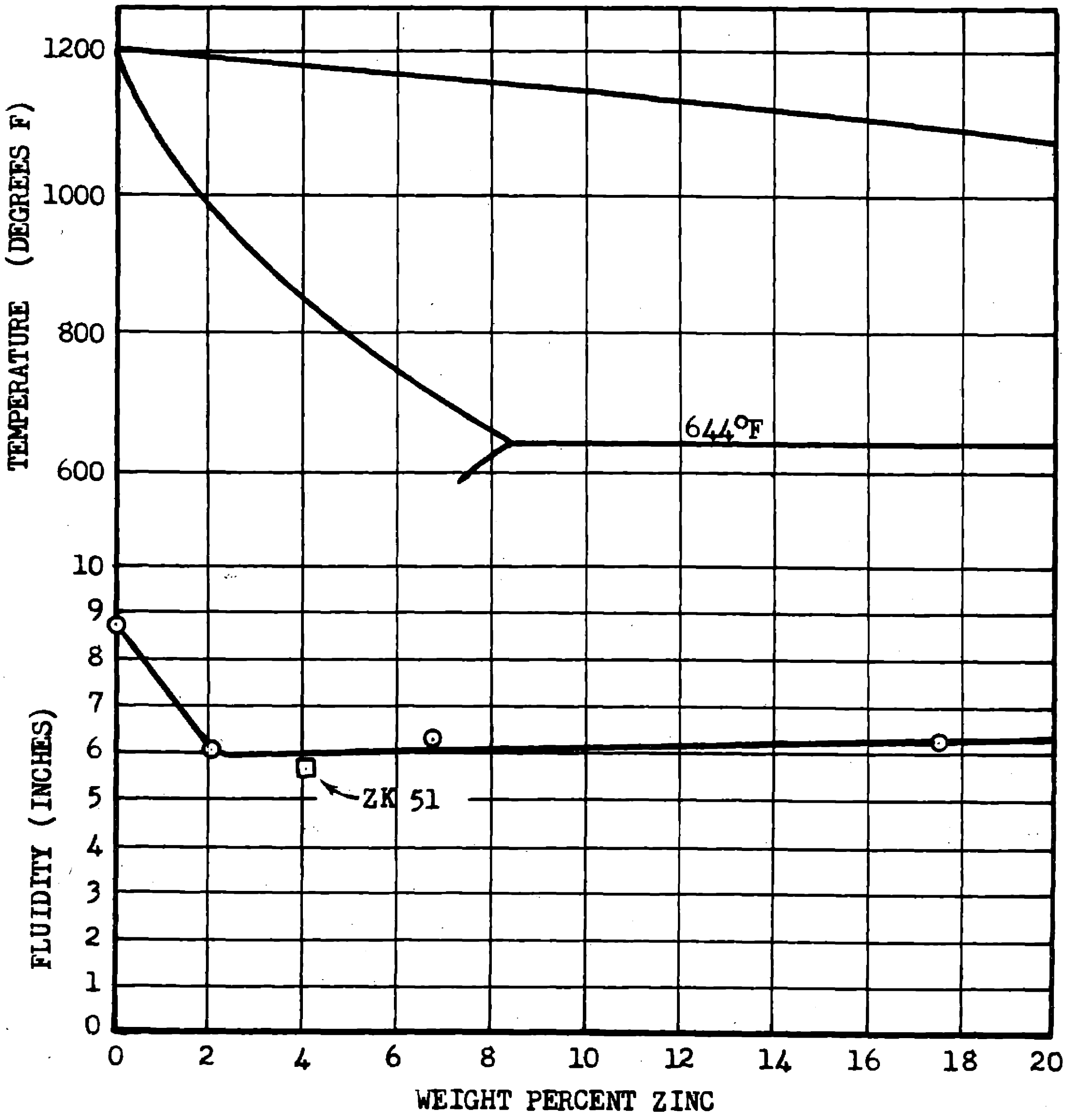

Figure 13 - Fluidity of Magnesium-Zinc-0.5% Zirconium Alloys at 1400°F from 0 to 20% Zinc.

the liquidus temperature is not known. However, the slopes of the curves in Appendix B indicate that the curves at constant superheat would be roughly parallel to the curve at 1400°F - similar to the fluidity curves for the magnesium-zinc system with no zirconium.

The general form of the fluidity curves of Figure 13 is similar to that of Figure 9 which contains fluidity curves for the magnesium-zinc system with no zirconium. As would be expected the fluidity of pure magnesium. However, when the zinc content of the magnesium-zirconium alloy is more than five percent, its fluidity is slightly greater than the corresponding zinc alloy with no zirconium. This slight increase may be caused by the effect of zirconium on the mode of solidification. Zirconium is a strong nucleating agent in these alloys, and all such alloys investigated microscopically revealed a very fine grained, equiaxed structure; the alloys with no zirconium showed a columnar structure.

Commercial alloy ZK51 was tested and its fluidity at 1400°F is included in Figure 13. The agreement with the experimental alloys is good.

MAGNESIUM-THORIUM-ZIRCONIUM SYSTEM

The fluidity of the magnesium-thorium-zirconium system as a function of thorium content is shown in Figure 14.

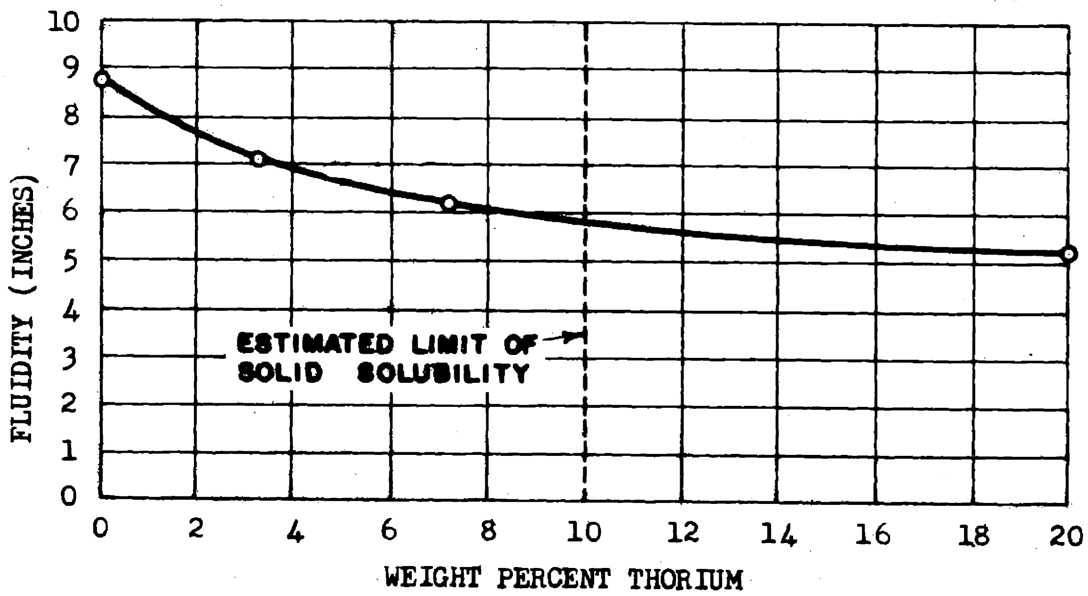

Figure 14 - Fluidity of Magnesium-Thorium-0.5% Zirconium Alloys at 1400°F from 0 to 20% Thorium.

Very little is known of the phase diagram of the magnesium-thorium binary system. Therefore, a curve of fluidity at constant superheat could not be drawn. The estimated limit of thorium solubility in pure magnesium is shown in Figure 14 at ten percent thorium.

The fluidity of the thorium alloys decreased significantly as thorium content increased above five percent thorium. This behavior is unusual especially since the estimated composition of the eutectic is only thirty-five percent thorium. No explanation of this behavior can be made at this time due to the limited available information on this system.

Figure 15 shows fluidity curves as a function of temperature for experimental alloy 80 and commercial alloy ZH62. Alloy 80 contained magnesium, 7.10 percent zinc, 1.62 percent thorium, and 0.91 percent zirconium; alloy ZH62 was analyzed after testing and contained 5.30 percent zinc, 1.23 percent thorium, and 0.69 percent zirconium.

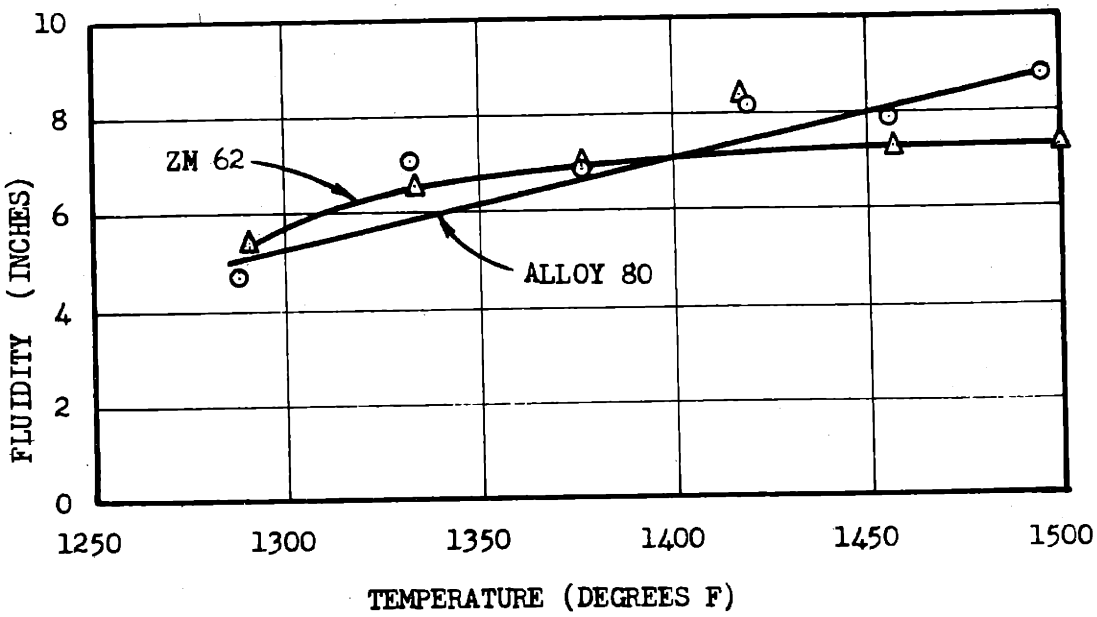

Figure 15 - Fluidity of Alloy 80 and Alloy ZH62 as a Function of Temperature.

MAGNESIUM-RARE EARTHS AND MAGNESIUM-RARE EARTHS-ZIRCONIUM SYSTEMS

The fluidity of the magnesium-rare earths-zirconium system at 1400°F is shown in Figure 16. Four points of the magnesium-rare earth system are also shown; however, in three of the alloys only a small amount of rare earths dissolved. These three points can only indicate a trend at very low rare earth content. The fluidity of magnesium with 8.70 percent rare earths is shown.

The immediate rise in fluidity with small additions of rare earths cannot be explained due to the limited available information on the

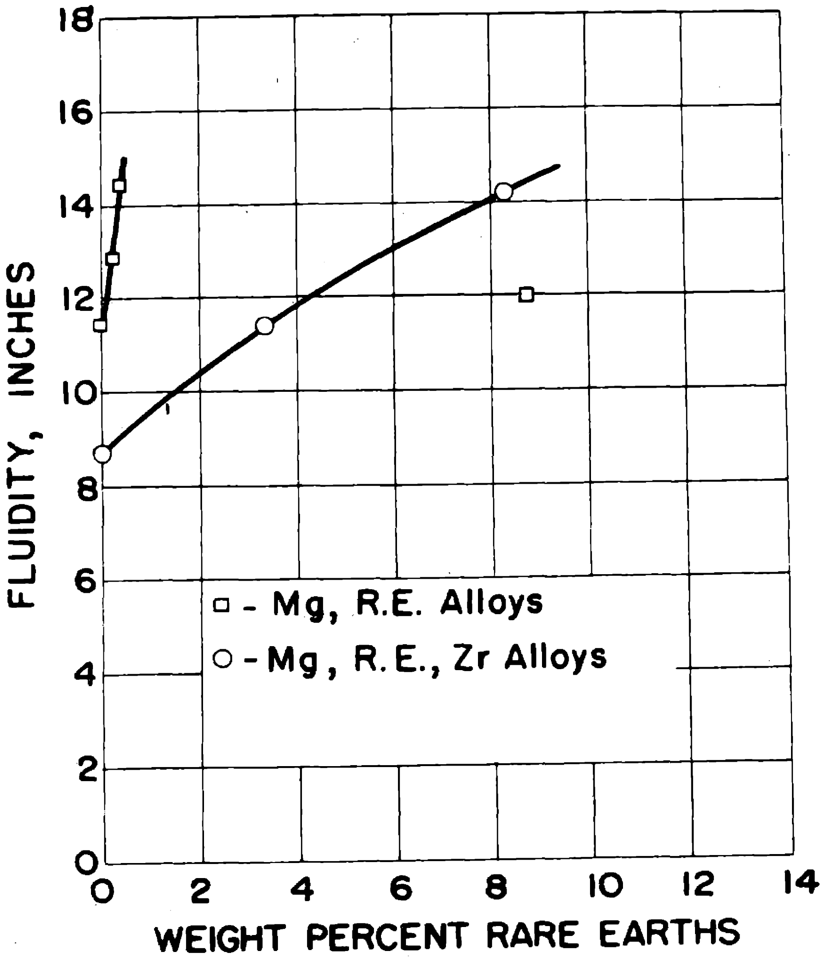

Figure 16 - Fluidity of Magnesium-Rare Earths and Magnesium-Rare Earths-Zirconium Systems at 1400°F from 0 to 9% Rare Earths.

magnesium-rare earths systems.

The major constitutents of Mischmetal (rare earths) are cerium and lanthanum. It is known that the maximum solubility of cerium in magnesium is about 1.6 percent cerium and that there is a eutectic at about twenty-one percent cerium.[34] The maximum solubility of lanthanum in magnesium is about 2.6 percent lanthanum and there is a eutectic at about 10.8 percent lanthanum.[35] However, this information does not, in itself, explain the fluidity curves.

Figure 16 shows that, at small rare earth content, the effect of zirconium is to decrease fluidity; but at higher rare earth content (eight percent), the zirconium addition increases fluidity. This behavior is similar to that detected in the magnesium-zinc-zirconium system. The zirconium, acting as a strong nucleating agent, prevents the formation of large grains. Thus, when a large fraction of the melt is solid and "mushy" type solidification is present, the mixture of small grains and liquid can still flow easily. When coarse grains are formed (absence of nucleating agent), the mixture of grains and liquid very shortly ceases to flow due to grain interference.

VI. CONCLUSIONS

1. The vacuum fluidity apparatus is a feasible means for the determination of fluidity of magnesium alloys.

2. In all magnesium alloy systems studied, except the rare earths systems, there was a sharp drop in fluidity as small amounts of alloying elements were added. When alloy content approached eutectic composition, a rise in fluidity was evident.

3. Adding zirconium to pure magnesium reduced the fluidity of magnesium. But additions of zirconium to magnesium alloys of over four percent zinc or rare earths increased the fluidity slightly. This effect is believed to be caused by the fact that zirconium is a strong nucleating agent and produces fine grains thus counteracting the effects of grain interference on the flow of a partially frozen metal stream.

4. In general the fluidity varied inversely as the freezing range, or the temperature difference between the liquidus and solidus. Non-equilibrium freezing range curves were computed for alloy systems for which phase diagrams had been established; the inverse of these curves followed the fluidity results much better than the inverse of the equilibrium freezing range curves.

5. The most fluid alloy encountered in this study was an alloy of magnesium containing 0.40 percent rare earths.

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VII. APPENDICES

APPENDIX A - Tabulation of Fluidity Data

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
Data: Magnesium-Aluminum Alloys			
32	Mg	1506	9.80
	1.4 Mn	1448	12.95
		1393	11.70
		1343	9.87
		1269	7.60
		1202	10.95
33	Mg	1505	9.25
	0.74 Al	1398	9.40
	1.4 Mn	1371	9.87
		1319	7.45
		1279	6.80
		1242	8.55
34	Mg	1503	5.70
	1.96 Al	1450	7.40
	1.4 Mn	1400	6.45
		1325	5.95
		1265	4.45
36	Mg	1505	6.60
	5,87 Al	1457	7.20
	1.4 Mn	1397	6.55
		1331	5.80
		1299	6.00
		1254	5.40
64	Mg	1514	7.60
	9.44 Al	1431	7.75
	1.2 Mn	1363	5.95
		1283	6.80
		1208	4.45
		1133	4.55
39	Mg	1494	7.12
	10.32 Al	1433	8.25
	1.2 Mn	1358	5.60
		1281	4.12
		1214	5.45
		1143	3.05

APPENDIX A - Tabulation of Fluidity Data (continued)

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
63	Mg	1502	8.45
	20.5 Al	1430	9.25
	1.2 Mn	1357	8.05
		1277	8.50
		1205	8.55
		1132	6.60
Data: Magnesium-Zinc Alloys			
41	Mg	1495	5.80
	2.64 Zn	1435	7.30
	1.4 Mn	1385	5.70
		1334	5.80
		1286	5.15
		1239	3.30
42	Mg	1506	7.85
	6.86 Zn	1442	4.85
	1.3 Mn	1383	5.05
		1327	5.45
		1275	4.55
		1221	2.60
43	Mg	1494	5.50
	13.46 Zn	1445	6.55
	1.2 Mn	1374	4.75
		1298	4.75
		1230	9.00
		1156	2.85
Data: Magnesium-Aluminum-Zinc Alloys			
44	Mg	1508	7.25
	8.49 Al	1421	6.60
	4.39 Zn	1369	5.15
	1.2 Mn	1351	5.30
		1300	4.95
		1249	3.20
45	Mg	1491	7.20
	8.49 Al	1464	6.60
	4.26 Zn	1405	6.25
	1.2 Mn	1350	6.55
		1297	5.85
		1252	5.55

APPENDIX A - Tabulation of Fluidity Data (continued)

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
48	Mg	1494	8.00
	8.12 Al	1410	8.05
	7.70 Zn	1338	7.05
	1.2 Mn	1273	6.20
		1203	5.70
		1128	4.00
51	Mg	1500	8.70
	12.90 Zn	1431	8.45
	3.79 Al	1351	6.50
	1.2 Mn	1274	5.25
		1188	5.70
		1117	3.80
52	Mg	1498	6.00
	3.85 Al	1446	8.60
	2.95 Zn	1379	6.10
	1.3 Mn	1329	5.25
		1275	5.75
		1219	4.85
55	Mg	1532	7.15
	10.08 Al	1475	7.45
	2.28 Zn	1395	6.80
	1.3 Mn	1321	6.05
		1254	5.87
		1191	3.25
58	Mg	1508	8.40
	11.00 Zn (estimate)	1425	7.30
	3.33 Al	1360	9.30
	1.3 Mn	1291	5.40
		1224	4.60
		1156	3.80

Data: Magnesium-Zinc-Zirconium Alloys

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
70	Mg	1483	9.45
	0.42 Zr	1438	8.40
		1403	11.10
		1357	8.70
		1310	8.05
		1268	7.87

APPENDIX A - Tabulation of Fluidity Data (continued)

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
69	Mg	1505	6.70
	2.09 Zn	1435	6.75
	0.21 Zr	1380	5.95
		1331	6.00
		1285	6.40
		1236	4.60
71	Mg	1480	7.00
	6.77 Zn	1436	8.70
	0.49 Zr	1373	6.30
	(0.58 Zr -chemical	1324	5.75
	analysis)	1272	4.80
		1217	4.75
73	Mg	1490	8.20
	17.62 Zn	1435	13.95
	0.34 Zr	1370	6.15
		1289	6.75
		1226	6.00
		1175	4.50

Data: Magnesium-Thorium-Zirconium Alloys

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
77	Mg	1485	8.50
	3.15 Th	1439	7.95
	0.48 Zr	1405	7.85
		1367	6.30
		1328	13.75
		1283	5.05
75	Mg	1486	7.60
	7.15 Th	1458	6.55
	0.56 Zr	1416	6.70
		1364	5.50
		1326	5.15
		1287	4.60
76	Mg	1503	6.10
	21.5 Th	1450	5.70
	0.84 Zr	1411	5.50
		1368	4.95
		1323	4.70
		1272	4.40

APPENDIX A - Tabulation of Fluidity Data (continued)

Alloy	Analysis	Temperature, Degrees Fahrenheit	Fluidity (inches)
Data:	Magnesium-Zinc-Thorium-Zirconium Alloy		
80	Mg	1495	8.75
	7.10 Zn	1456	7.85
	1.62 Th	1419	8.20
	0.91 Zr	1376	6.85
		1332	7.05
		1288	4.70
Data:	Magnesium-Rare Earth-Zirconium Alloys		
82	Mg	1485	12.90
	3.30 R. E.	1440	10.45
	0.35 ZR	1407	10.40
		1349	10.50
		1313	12.00
		1279	9.30
83	Mg	1506	16.60
	8.25 R. E.	1461	17.45
	0.26 Zr	1412	15.80
		1376	12.50
		1334	13.65
		1285	10.80
84	Mg	1492	14.40
	16.50 R. E.	1434	11.60
	0.18 Zr	1400	11.55
		1348	9.60
		1313	8.50
		1273	6.95
Data:	Magnesium-Rare Earth Alloys		
85	Pure Mg	1496	11.95
		1462	11.85
		1418	12.45
		1374	11.95
		1333	10.80
		1295	10.75

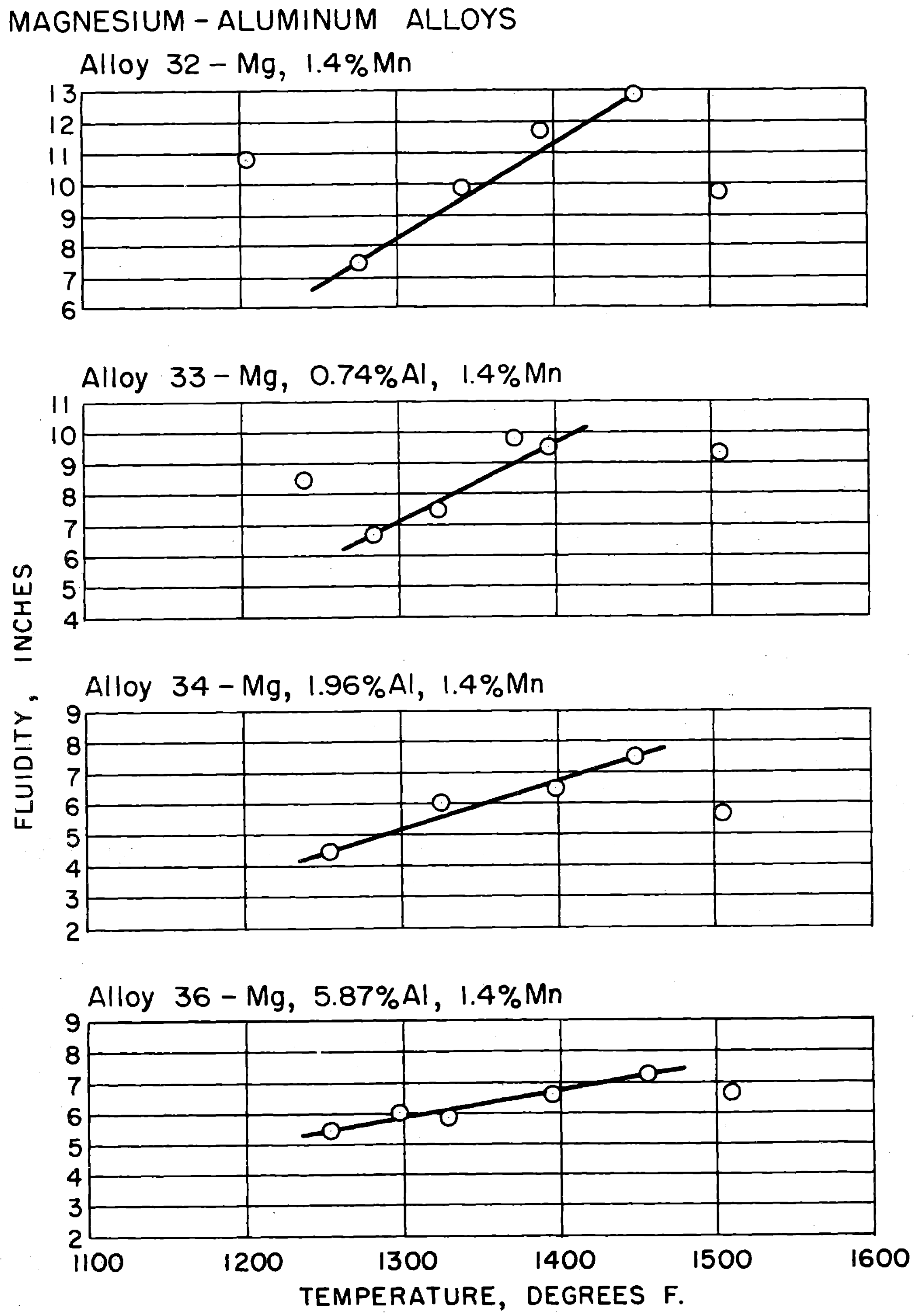
APPENDIX A - Tabulation of Fluidity Data (continued)

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
86	Mg	1478	14.95
	0.20 R. E.	1443	14.95
		1400	12.10
		1355	13.70
		1317	10.60
		1278	9.80
88	Mg	1495	18.85
	0.40 R. E.	1452	14.80
		1406	15.85
		1339	12.60
		1287	9.30
		1260	10.25
89	Mg	1503	15.05
	8.70 R. E.	1461	13.85
		1411	12.80
		1372	15.80
		1318	9.40
		1266	4.55

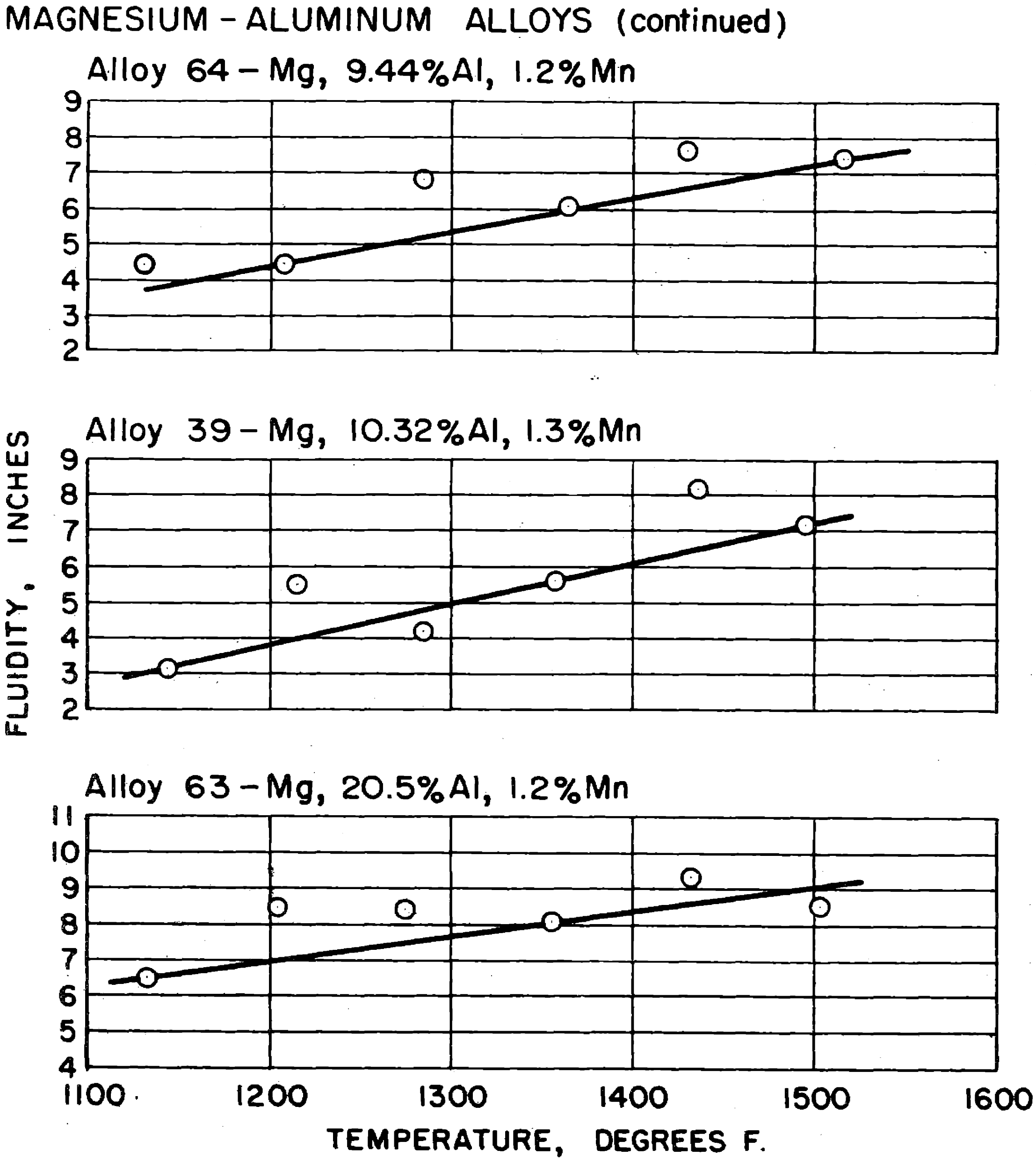
Data: Commercial Alloys

Alloy	Analysis	Temperature Degrees Fahrenheit	Fluidity (inches)
AZ92	Mg	1495	6.40
	8.90 Al	1440	7.10
	2.10 Zn	1379	5.80
	0.23 Mn	1311	6.75
		1230	9.05
		1153	5.05
ZK51	Mg	1486	6.00
	4.10 Zn	1436	7.75
	0.10 Zr	1371	5.45
		1305	5.55
		1238	4.80
		1181	3.05
ZH62	Mg	1506	7.30
	5.30 Zn	1457	7.30
	1.23 Th	1417	8.40
	0.69 Zr	1376	6.95
		1333	6.55
		1291	5.30

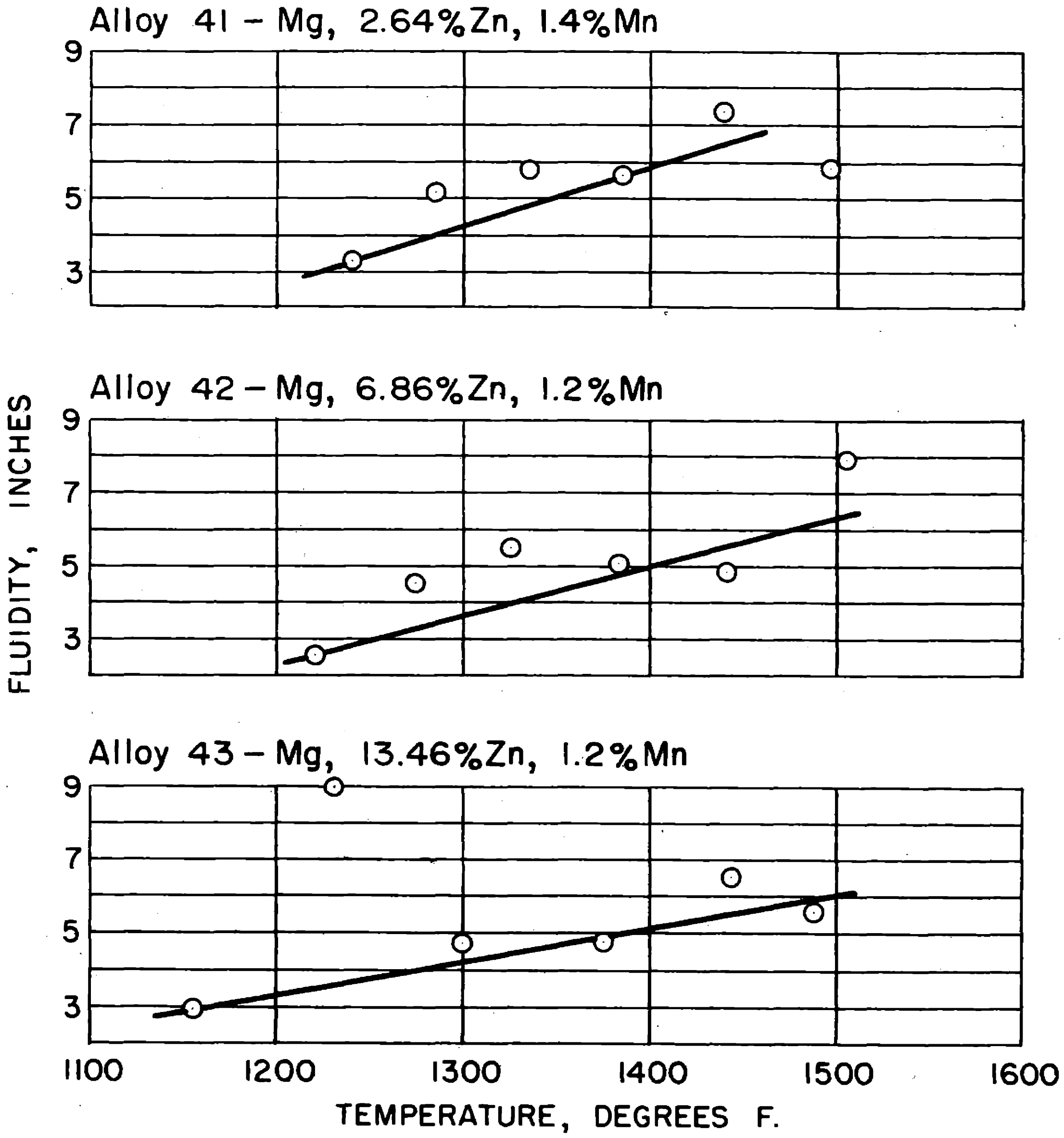
APPENDIX B - Curves of Fluidity versus Temperature.



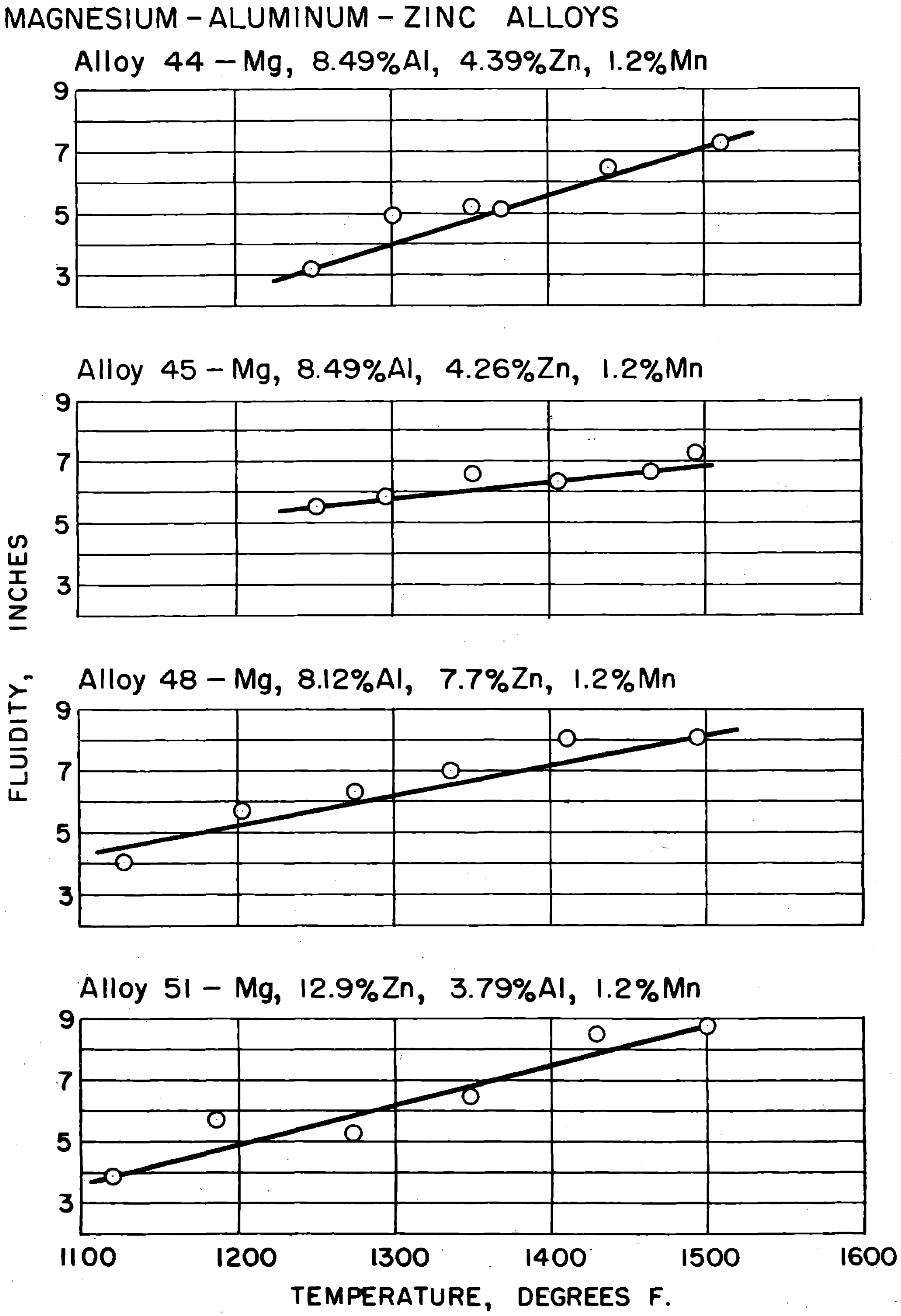
APPENDIX B - Curves of Fluidity versus Temperature (continued)



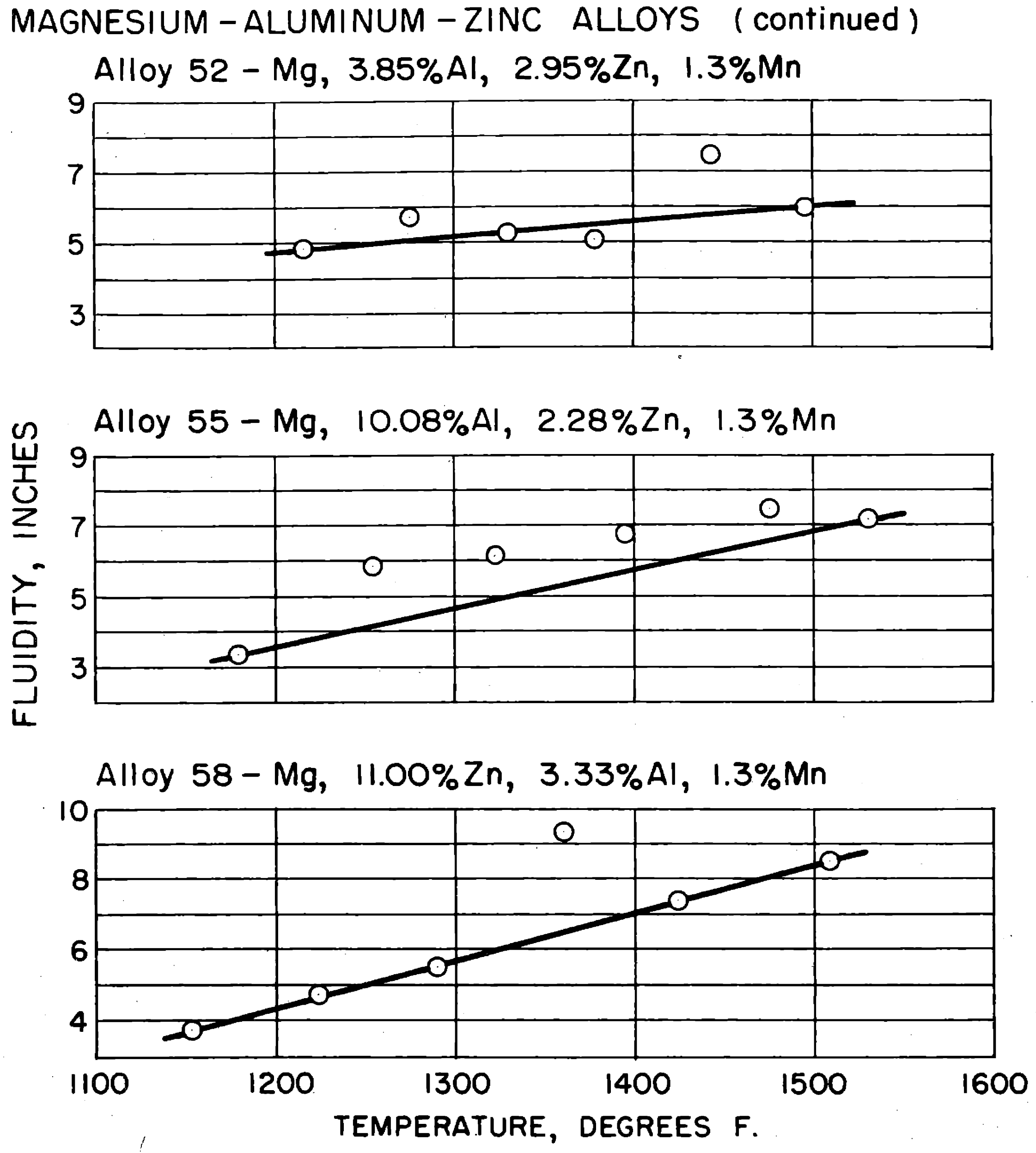
MAGNESIUM - ZINC ALLOYS



APPENDIX B - Curves of Fluidity versus Temperature (continued)



APPENDIX B - Curves of Fluidity versus Temperature (continued)



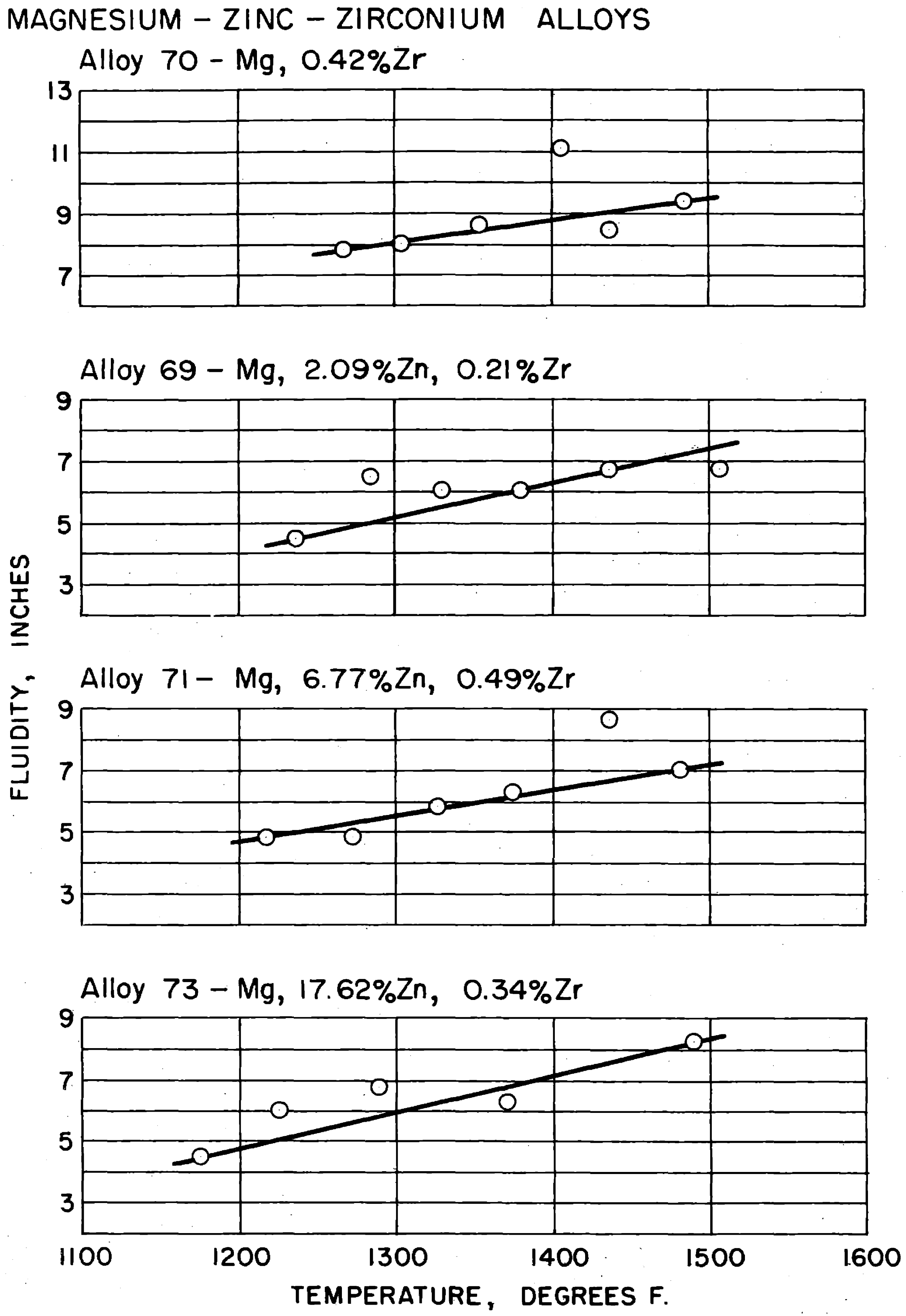
MAGNESIUM - ALUMINUM - ZINC ALLOYS (continued)

Alloy 52 - Mg, 3.85%Al, 2.95%Zn, 1.3%Mn

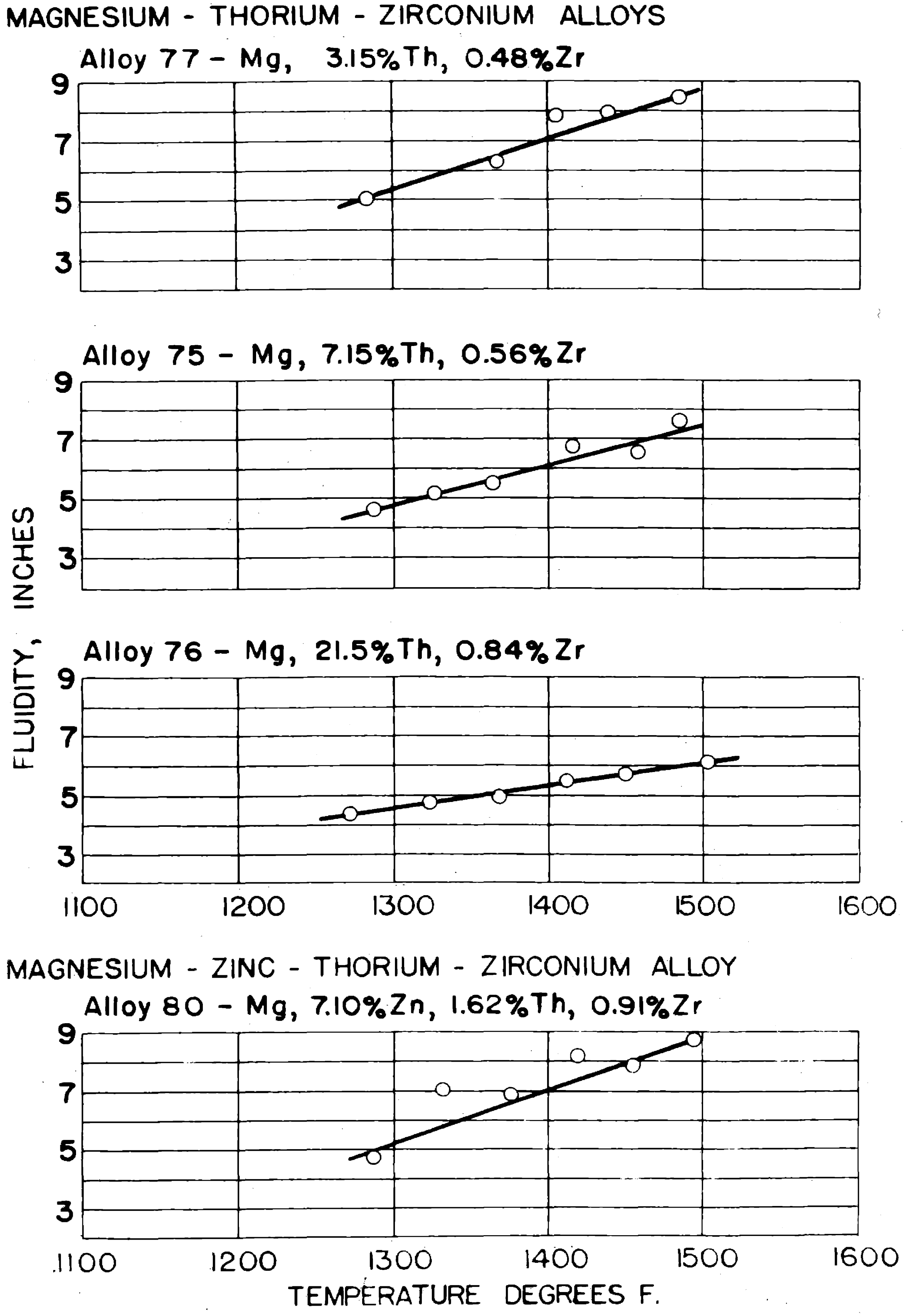
Alloy 55 - Mg, 10.08%Al, 2.28%Zn, 1.3%Mn

Alloy 58 - Mg, 11.00%Zn, 3.33%Al, 1.3%Mn

APPENDIX B - Curves of Fluidity versus Temperature (continued)

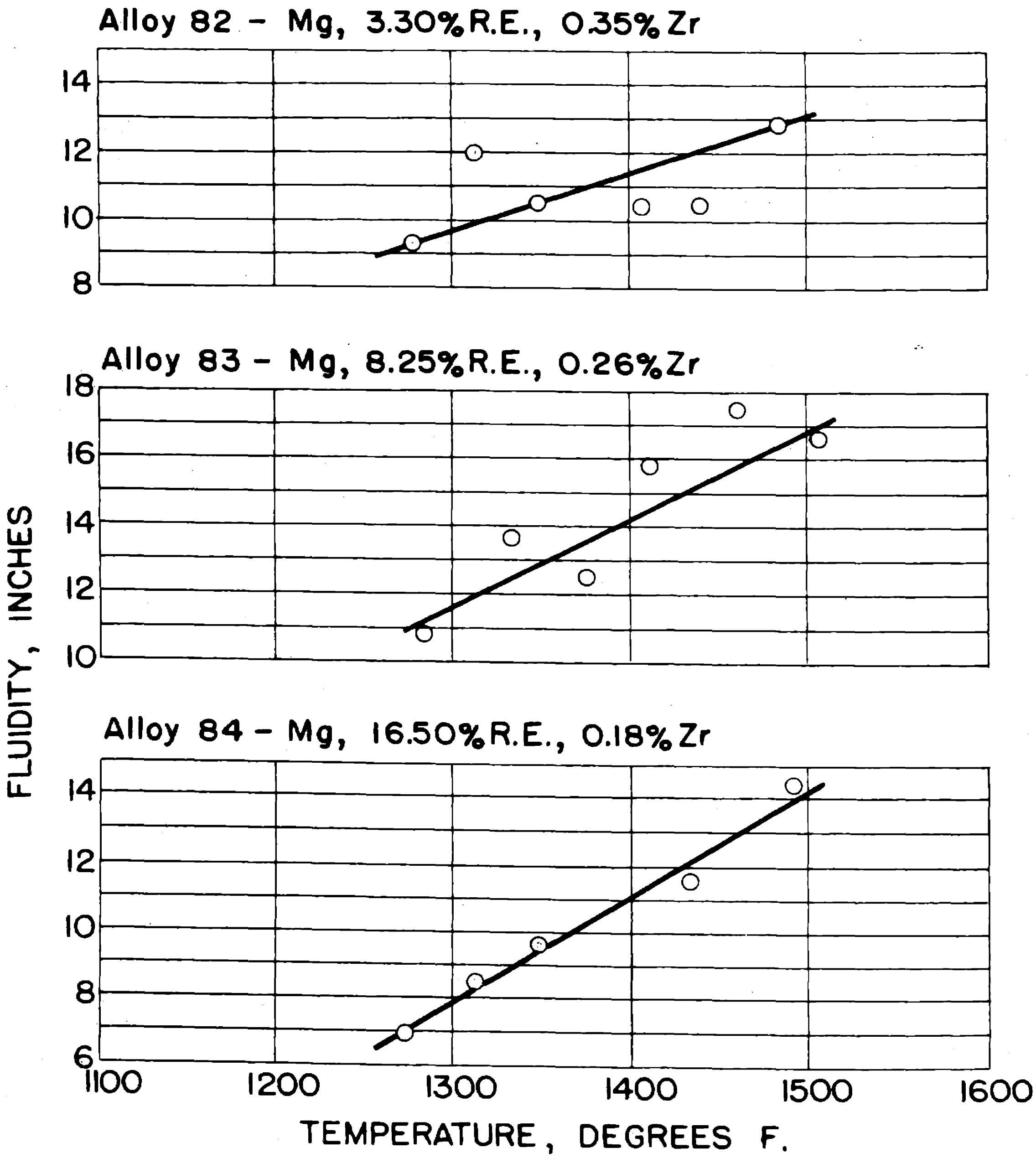


APPENDIX B - Curves of Fluidity versus Temperature (continued)



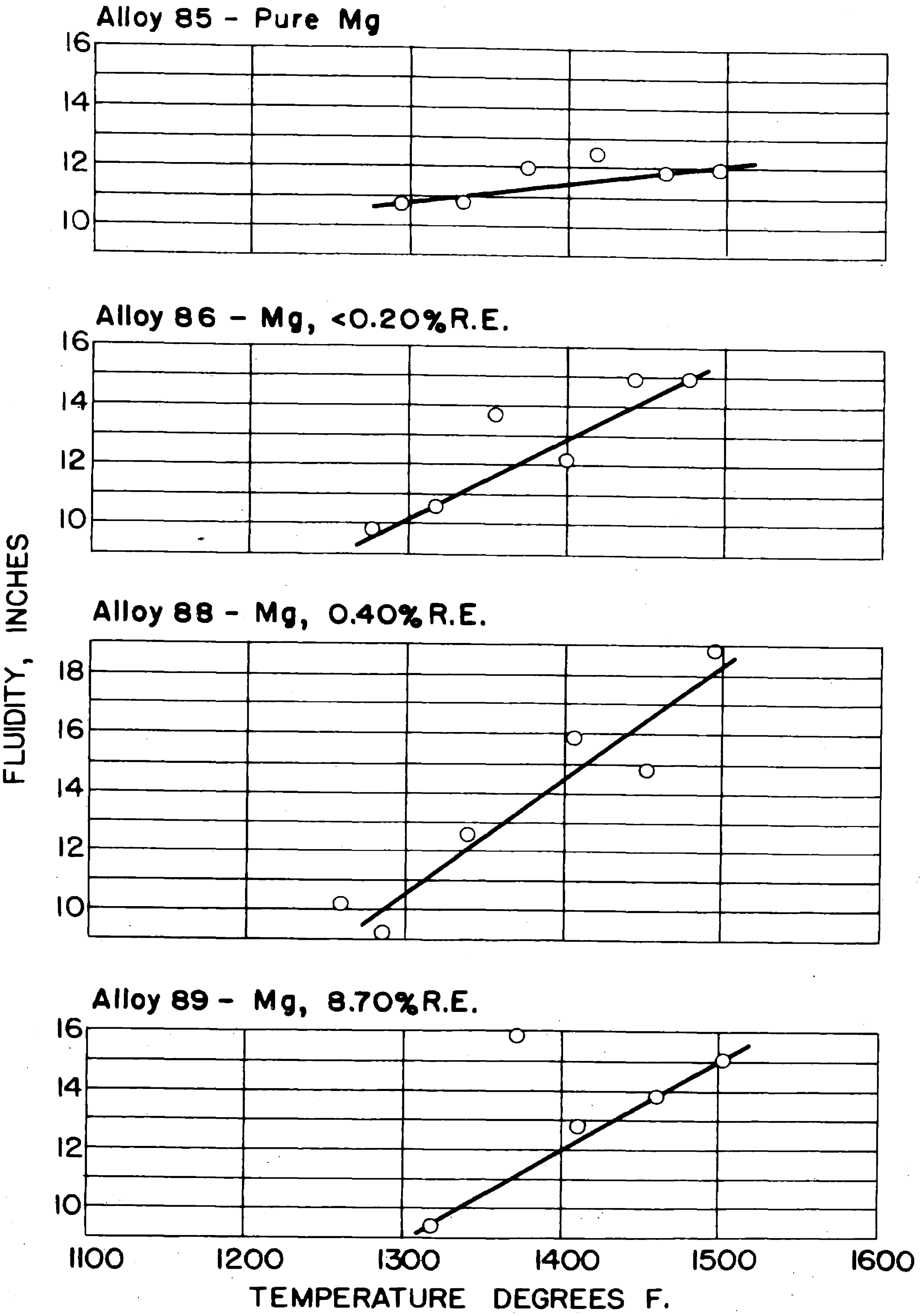
APPENDIX B - Curves of Fluidity versus Temperature (continued)

MAGNESIUM - RARE EARTH - ZIRCONIUM ALLOYS



APPENDIX B - Curves of Fluidity versus Temperature (continued)

MAGNESIUM - RARE EARTH ALLOYS



Alloy 85 - Pure Mg

Alloy 86 - Mg, <0.20% R.E.

Alloy 88 - Mg, 0.40% R.E.

Alloy 89 - Mg, 8.70% R.E.

APPENDIX B - Curves of Fluidity versus Temperature (continued)