Alexandria Division

AMERICAN MACHINE & FOUNDRY COMPANY

DEVELOPMENT OF
LIGHT WEIGHT MAGNESIUM ALLOYS
FOR LOW TEMPERATURE APPLICATIONS

Annual Progress Report
June 19, 1965 through March 20, 1966

Contract No. NAS 8-11168
DCN-1-4-50-01030-01(IF)

March 20, 1966

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ACKNOWLEDGEMENT

ABSTRACT

The development of light weight magnesium alloys for cryogenic applications continued through the present reporting period which ran from June 19, 1965 to March 20, 1966. As this period began one alloy, designated II 4, appeared promising for further development. Its ultimate tensile strength was close to the goal of 45,000 psi, its yield strength was greater than 35,000 psi, it was ductile but percent elongation values were very sensitive to prior thermal-mechanical treatment. On the debit side, the alloy was notch sensitive, appeared difficult to weld, and reached optimum mechanical properties after very short time aging, one hour and twenty minutes, at 200°F. One other alloy, IA 6, which was a modification of alloy II 4 containing 9% Li and 2% Zn + 4% Al with a small addition of Mn instead of the almost 1:1 ratio of 7%Li + 6% Zn for alloy II 4, had very high strength and good ductility at -452°F but a tensile strength of only 25,000 psi at ambient temperature.

Further modifications were made to the composition of alloy II 4 during the present reporting period which led to the development of an alloy designated ZLH 972. The average ultimate tensile strength of this alloy was 44,100 psi, its average yield strength (0.2% offset) was 37,600 psi, and its average elongation (within 2") was 18.4%. The alloy was not notch sensitive and it was easy to weld.

The stability of alloy II 4 was improved to the point where it became necessary to age it for twenty hours at 200°F to achieve the optimum compromise between strength and ductility. With the development of this new heat treatment the alloy was no longer notch sensitive and its weld efficiency was raised to values greater than 90%.

The most outstanding achievement reported herein is the raising of the ultimate tensile strength of alloy IA 6 from 25,000 psi to an average value of approximately 42,000 psi, with an average yield strength of 40,000 and elongation (within 2") of 17.1%. These properties were obtained by optimizing the prior thermal-mechanical history of the alloy. In addition to good mechanical properties at both ambient and cryogenic temperatures, the alloy was not notch sensitive and could be welded easily without cracking. However, additional post-weld heat treating studies will be required to fully develop the alloy because consistent weld joint efficiencies of 80% or greater could not be obtained.

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1.0 INTRODUCTION

The purpose of this program is to develop a wrought magnesium alloy or alloys with superior mechanical properties at ambient and cryogenic temperatures. Specific mechanical and fabricability properties required as contractual goals for the alloy(s) include:

(1) A tensile strength of 45,000 psi, a yield strength (0.2% offset) of 35,000 psi, and 20% elongation at room temperature (68°F); no deterioration of these properties is desired to -423°F (-253°C) except that the developed alloy(s) may have a minimum of 15% elongation;

(2) A notch/unnotched tensile ratio of at least 1.0 at ambient temperature, with a goal of 0.90 at -423°F;

(3) Weldability by fusion methods equal to that of AZ 31B magnesium alloy;

(4) Capability for chemical conversion; anodic, or other suitable protective coating application .

A matrix alloying plan, using addition elements selected on the basis of present day alloying concepts was employed to obtain compositions with the stated property and fabrication characteristics. The major alloying concepts utilized here include the following:

(1) Solid solution strengthening, as predicted by applying fundamental alloying rules, i.e., atomic size, compressibility and valency relations;

(2) Formation of intermetallic compounds for one or more of the following effects: (a) decrease solubility with temperature to secure favorable precipitation hardening, (b) crystallographic coherency with either a hexagonal or body-centered solid solution matrix to promote strengthening by suitable precipitation and/or optimal spheroidizing heat treatment, (c) discontinuous grain boundary precipitation of Laves and related intermetallic phases to improve hot strength without impairing hot-rolling plasticity needed for successful sheet-forming and welding;

(3) Nucleant-type grain refining to improve homogeneity and resulting mechanical properties and metal-working characteristics.

1.1 Program Planning

The results of work performed during a nine-month continuation to Contract No. NAS 8-11168, which originally began on June 19, 1964, are presented in this Summary Report which covers the period from June 19, 1965 to March 20, 1966. A chart reflecting the entire program schedule and planning is shown in Figure 1 on the following page. The major difference between this chart and the one presented at the beginning of the continuation program is an extension of the "Preparation of 50-pound Pilot Melts" to the end of December. This was done so that knowledge gained in preparing the first 50-pound melts could be applied to making cleaner, dross-free ingots. Modifications were made to the melting crucible and to the permanent mold so that additional ingots could be made of all three alloys chosen for scale-up operations. An effort was made to get the program back on its original schedule by performing a survey type testing program, i.e., single or duplicate tests were performed on each alloy under a wide variety of thermal-mechanical conditions. The major testing program, which consisted of testing several identical specimens prepared under what was determined from survey tests to be optimum rolling and heat treating conditions, was performed at the end of the program. The extra ingots made in December were used for additional rolling studies. As explained in the text, this change in scheduling to accommodate additional studies paid dividends because a thermal-mechanical treatment was discovered which increased the ambient temperature strength of one of the alloys by 80%.

TASKS	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
Grain Size Studies		▬								
Preparation of 10 Pound Castings		▬▬	▬▬	▬ ▬	▬ ▬					
Mechanical Property Screening Tests			▬▬	▬▬	▬▬	▬				
Welding Studies				▬▬ ▼			▬ ▬	▬ ▬		
Cryogenic Testing of 10 Pound Castings						▬▬	▬			
Preparation of 50 Pound Pilot Melts				▬	▬ ▬	▬▬	▬			
Testing of Pilot Ingot Material							▬ ▼	▬		
Cryogenic Testing of Pilot Ingot Material									▬	
Coatings – Application and Testing							▬ ▬		▬	
Metallography and X-Ray Diffraction			▬▬▬▬▬▬▬▬▬▬▬▬▬▬▬▬▬▬							

Reporting:

	June	July	Aug.	Sept.	Oct.	Nov.	Dec.	Jan.	Feb.	March
Monthly		▬	▬	▬		▬	▬		▬	◄
Quarterly					▬			▬		
Final									▬	▬

CODE: ▼ Cryogenic Liquid Required At This Time

Figure 1.   Task Schedule

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2.0 TECHNICAL REVIEW OF WORK ACCOMPLISHED DURING THE PERIOD FROM JUNE 19, 1965 THROUGH MARCH 20, 1966

During the original twelve-month program, which began on June 19, 1964, forty magnesium alloys were melted and cast in a cast iron permanent mold. The compositions of these alloys were selected according to a statistical plan in order to cover as wide a range of strength and ductility values as possible. Three alloying systems were originally selected and each system was represented by a statistical matrix containing nine alloying compositions as shown in Figure 2 on the following page. None of the alloys in matrix I came close to meeting the ambient temperature strength requirements of the program so this alloy system was modified as shown in Matrix IA, Figure 3. The most promising of these compositions was alloy II 4 (Mg - 1 Zr - 3 Th - 5 Cd - 6 Zn - 6 Ag - 7 Li) which had a tensile strength that varied from 36,000 psi to 51,000 psi at ambient temperature depending upon prior thermal-mechanical treatments. The remaining alloys studied during the course of this original program were cadmium and thorium variations of alloy II 4.

2.1 Selection of Alloying Compositions for the Preliminary Melts

The primary objective for the first three months of the present reporting period was to prepare for the initial scale-up melting operation in October 1965. The alloy to be chosen for the initial scale-up melt was to be made by mutual agreement between the NASA project manager and AMF project personnel. This decision was made after an evaluation of additional refinements to alloy II 4 and examining alloys containing from 7 to 14% lithium with various amounts of aluminum, zinc, and silver, plus a further evaluation of magnesium-9% lithium alloys with various amounts of manganese, aluminum, zinc, and silver. The composition of these preliminary melts is shown in matrices IV, V, and VI in Figure 4.

The original research program was based upon a plan of approach that required melting and casting all the alloys in each matrix for screening purposes and then to modify the composition of the most promising alloy for final optimization of mechanical properties. This approach worked well for matrix II and matrix IA and a good comparison was made between the properties of these alloys. On the other hand, a great deal of time was spent on fabrication studies of 18 alloys from matrix I and matrix III without promising results. In order to eliminate time wasted in processing alloys with little chance of meeting the contractual goals, the plan of approach was changed slightly. In the new approach, three alloys were selected from each matrix for processing, and, based upon the mechanical properties of these three alloys, the remaining alloys in the matrix were either eliminated from the program, processed as originally planned, or modified in composition to incorporate the latest scientific alloying principles developed during the course of the program.

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MATRIX I

Basic Alloy - Pure Mg

	0.05 Li	0.2 Li	1.0 Li
.25 Mn	1 3 Al 6 Zn	2 8.5 Al 3 Zn	3 12 Al 1 Zn
.75 Mn	4 12 Al 3 Zn	5 3 Al 1 Zn	6 8.5 Al 6 Zn
1.5 Mn	7 8.5 Al 1 Zn	8 12 Al 6 Zn	9 3 Al 3 Zn

MATRIX II

Basic Alloy - Mg + 1 Zr + 3 Th

	5 Cd	10 Cd	20 Cd
2 Zn	1 2 Ag 2 Li	2 4 Ag 7 Li	3 6 Ag 12 Li
6 Zn	4 6 Ag 7 Li	5 2 Ag 12 Li	6 4 Ag 2 Li
12 Zn	7 4 Ag 12 Li	8 6 Ag 2 Li	9 2 Ag 7 Li

MATRIX III

Basic Alloy - Mg + 1 Zr

	0 Nd	1.5 Nd	3 Nd
0 Th	1 2 Zn 2 Ag	2 6 Zn 4 Ag	3 9 Zn 6 Ag
3 Th	4 9 Zn 4 Ag	5 2 Zn 6 Ag	6 6 Zn 2 Ag
5 Th	7 6 Zn 6 Ag	8 9 Zn 2 Ag	9 2 Zn 4 Ag

Figure 2.   The Original Statistical Alloying Plan for Selecting Alloying Compositions

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MATRIX I A

Basic Alloy - Mg - 3 Th - 9 Li

	$\frac{1}{4}$ Mn	$\frac{1}{2}$ Mn	1 Mn
1 Zn	1 0 Al 4 Ag	2 4 Al 2 Ag	3 2 Al 1 Ag
2 Zn	4 2 Al 2 Ag	5 0 Al 1 Ag	6 4 Al 4 Ag
4 Zn	7 4 Al 1 Ag	8 2 Al 4 Ag	9 0 Al 2 Ag

Figure 3.   Modification of Matrix I

MATRIX IV

Basic Alloy Mg + 2 Th

	1 Al	3 Al	6 Al
3 Zn	1 1 Ag 12 Li	2 2 Ag 9 Li	3 0 Ag 7 Li
5 Zn	4 0 Ag 9 Li	5 1 Ag 7 Li	6 2 Ag 12 Li
7 Zn	7 2 Ag 7 Li	8 0 Ag 12 Li	9 1 Ag 9 Li

MATRIX V

Basic Alloy Mg + 2 Th + 9 Li

	$\frac{3}{4}$ Mn	$\frac{1}{2}$ Mn	$\frac{1}{4}$ Mn
0 Zn	1 1 Ag 3 Al	2 2 Ag 4.5 Al	3 0 Ag 6 Al
2 Zn	4 0 Ag 4.5 Al	5 1 Ag 6 Al	6 2 Ag 3 Al
4 Zn	7 2 Ag 6 Al	8 0 Ag 3 Al	9 1 Ag 4.5 Al

MATRIX VI

Basic Alloy Mg + 2 Th

	8 Li	12 Li	14 Li
2 Al	1 6 Cd 6 Ag	2 12 Cd 4 Ag	3 2 Cd 2 Ag
4 Al	4 2 Cd 4 Ag	5 6 Cd 2 Ag	6 12 Cd 6 Ag
6 Al	7 12 Cd 2 Ag	8 2 Cd 6 Ag	9 6 Cd 4 Ag

Figure 4. Preliminary Alloying Compositions for the Preparation of 50-Pound Melts

The alloys from matrix IV, matrix V and matrix VI selected for initial testing were:

IV 2   (Mg - 2 Th - 2 Ag - 3 Al - 9 Li - 3 Zn)

IV 7   (Mg - 2 Th - 2 Ag - 1 Al - 7 Li - 7 Zn)

IV 9   (Mg - 2 Th - 1 Ag - 6 Al - 9 Li - 7 Zn)

V 6   (Mg - 2 Th - 2 Ag - 3 Al - 9 Li - 2 Zn - 0. 25 Mn)

V 7   (Mg - 2 Th - 2 Ag - 6 Al - 9 Li - 4 Zn - 0. 75 Mn)

V 8   (Mg - 2 Th - 0 Ag - 3 Al - 9 Li - 4 Zn - 0. 50 Mn)

VI 2   (Mg - 2 Th - 4 Ag - 2 Al - 12 Li - 12 Cd)

VI 5   (Mg - 2 Th - 2 Ag - 4 Al - 12 Li - 6 Cd)

VI 8   (Mg - 2 Th - 6 Ag - 6 Al - 12 Li - 2 Cd)

The refinements to alloy II 4 made prior to selecting the first alloy for scale-up melting consisted of the following modifications to the basic composition:

II 4   Mg + 1 Zr + 3 Th + 6 Zn + 5 Cd + 6 Ag + 7 Li

II 4 A   Mg + 1 Zr + 2 Th + 6. 3 Zn + 5 Cd + 4. 85 Ag + 9 Li

II 4 B   Mg + $C_6 Cl_6$ + 2 Th + 6 Zn + 2. 5 Cd + 6 Ag + 8 Li

II 4 C   Mg + $C_6 Cl_6$ + 2 Th + 6 Zn + 5 Cd + 0 Ag + 9 Li

II 4 D   Mg + $C_6 Cl_6$ + 2 Th + 8 Zn + 0 Cd + 0 Ag + 7 Li

II 4 E   Mg + 0 + 2 Th + 6 Zn + 5 Cd + 0 Ag + 9 Li

Alloy II 4 A was a repeat of a modification to alloy II 4 that was developed earlier in the program which showed good strength and elongation values. Alloys II 4 B, C and D were designed to show the effect of varying the solute concentrations, with special emphasis placed upon eliminating silver from the family of alloys. Alloy II 4 E was selected to show the effect of eliminating hexachlorobenzene from alloy II 4 C, a 9% lithium alloy.

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2.2 Preliminary Melting and Casting

One of the important discoveries of the initial program was the fact that neither zirconium or commercial grade silicon could effect grain refinement in magnesium alloys that contain more than 7% lithium. Chill casting was also not effective in producing a fine grain structure for alloys containing a large percentage of lithium. Since the usual techniques for grain refining magnesium alloys were found to be inadequate, the first melting and casting tasks performed in the current reporting period were made to evaluate the effect of small additions of hexachlorobenzene.

Various amounts of hexachlorobenzene were added to subsequent preliminary melts of the matrix IV and matrix V alloys just prior to casting. The resultant castings were characterized by a uniform, fine-grain structure and the addition of three grams of hexachlorobenzene per ten pounds of melt appeared to be optimum. Typical macrographs of fracture bars are shown in Figure 5 on the following page.

Refinements were also made to the small melting crucibles by increasing the height of the crucible above the melt, in order to provide for a greater volume of cover gas over the molten metal, and by fitting asbestos gaskets to the flanges for an improved gas-tight seal. A picture of the initial crucible design is shown in Figure 6 and the modified design is shown in Figure 7. The strip chart recorders and temperature controllers appear in the background.

As soon as high-lithium alloy castings could be made free of dross with the redesigned mold, the preparation of preliminary 10-pound castings was started. Earlier in the program manganese was added to the melt as a dusting flux and poor recovery values made an assessment of ensuing tensile data difficult. In order to improve the recovery values, a Mg - 25% Mn master alloy was made by the Belmont Smelting and Refining Company at the request of AMF project personnel. A casting was then made at AMF of an alloy containing 0.5% manganese, alloy V 8. Sections were cut from the top, center, and bottom of this casting and a complete analysis was made at the George C. Marshall Space Flight Center. The manganese recovery was: top - 0.39%, center - 0.40%, and bottom - 0.42%. These recovery values are considered to be excellent and conclusions concerning the effect of manganese can be made with confidence.

The 10-pound melts were cast into the permanent mold shown in Figures 8 and 9. The preliminary melts of magnesium-lithium alloys that contained a large percentage of aluminum appeared to be sluggish and the castings did not completely fill the mold. A fracture of each casting runner revealed some dross inclusions in these first castings. To alleviate the dross problem the mold sump was enlarged to accommodate a larger pair of

Fracture of alloy V-6 bar made with an addition of 2g. of hexachlorobenzene per 10 lb. of melt. The melt was single screened and the dark particles are dross. Note the fine grained texture of the fracture which is shown at 5X.

Fracture of alloy V-7 bar made with an addition of 3g. of hexachlorobenzene per 10 lb. of melt. The melt was double screened and the dark areas are holes caused by the pulling out of material during fracture. The very fine grained texture of the fracture is shown at 5X.

Fracture of alloy IV-7 bar made with an addition of 3g. of hexachlorobenzene per 10 lb. of melt. The melt was not screened and the large dark areas are dross. The fine grained texture of the fracture is shown at 5X.

Figure 5.

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Figure 6   Melting Crucible and Top Showing Entrance Port for Adding
Alloying Elements to the Melt

Figure 7. Melting Crucibles

The small melting crucibles are shown protruding from electric furnaces.
Each crucible has been increased in height to provide for a greater melting
volume and each top is tightly sealed to protect the melt from oxidation.

Figure 8. Design of Permanent Casting Mold showing initial dimensions of the sprue and runner. A bottom ingate was added after trial castings. Note: The side tapers are exagerated in the drawing for illustration.

Figure 9. Opened View of Cast Iron Permanent Mold Showing An Enlarged Runner And The Final Ingate Design. The Side Gate Was Blocked Off For Most Of the Melts And Inserts Were Used In The Cavity To Vary The Size Of The Preliminary Ingots For Research Purposes.

concentric screens and recessed to prevent any molten metal from bypassing the screens. The runner diameter was also increased slightly for improved fluidity.

The quality of the matrix VI alloys was not good because of the poor fluidity of these 12% lithium alloys. In order to develop these alloys a new mold would have to be designed and built incorporating a larger sprue-to-runner-to-ingate ratio. This did not seem either technically or economically advisable and the alloys were not processed further.

2.3 Processing the Ten-Pound Melts

All the castings of the matrix IV and V alloys successfully hot worked into 0.10 sheet. Hot working of these alloys was accomplished by heating the castings in a neutral salt bath at 800°F and reducing the cross sectional area 15% during each pass through the rolling mill. Cross rolling and unidirectional rolling were studied for at least one casting of each composition. Subsequent heat treating studies and rolling studies on similar alloys indicated that these materials are more ductile at lower temperatures and greater reductions might have been possible at such temperatures.

2.3.1 Metallography of the Matrix IV and Matrix V Alloys

For discussion purposes the as-cast structures of the initial six alloys of matrices IV and V are compared with their as-rolled structures in Figures 10 through 21. All of the as-rolled photomicrographs show the effect of cross rolling. Cross rolling alloy IV 2 completely broke up the elongated β phase in the as-cast structure into the lamellar structure shown in Figure 11 which turned out to be the most brittle as-rolled structure tested during the program. The as-cast structure of alloy IV 7 shows an unusually large amount of the ductile β phase for an alloy that contains only 7% lithium. Hot working at 800°F greatly reduced the amount of the ductile β phase shown in the as-cast photomicrograph in Figure 13 and explains, in part, why this alloy became harder after a high temperature heat treatment, although, at the same time, 30% elongation was obtained in the as-rolled condition. The as-cast and as-rolled structures of alloy IV 9 are compared in Figures 14 and 15. The ductile β phase was broken up by hot working and elongated equally in both directions of rolling. This type of structure resulted in good low temperature ductility in previous specimens, but alloy IV 9 was very brittle at -320°F. This surprising lack of ductility must have been caused by a brittle matrix because no brittle precipitates are apparent in the bcc β phase. Note that uniform black particles can be seen throughout the as-cast structure of alloy IV 9. These particles were not caused by hexachlorobenzene. The size of the particles in Figure 14 appear to be loo large for nucleant grain refinement and appear to be dispersed throughout the matrix and the β phase.

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Figure 10   250 X   Figure 11   250 X
Alloy IV 2   As Cast   Alloy IV 2   As Rolled

The lamellar structure shown in Figure 11 is brittle resulting in no elongation in the as-rolled condition. Solution heat treating this structure at 800°F and aging at 200°F raised the elongation to 17% but also reduced its tensile strength.

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| Figure 12 | 250 X | Figure 13 | 250 X |
| Alloy IV 7 | As Cast | Alloy IV 7 | As Rolled |

The large amount of β phase shown in Figure 12 is unusual for an alloy that contains only 7% lithium. This second phase was broken up by hot working at 800°F as shown in Figure 13 and an elongation value of 30.6% was obtained for a specimen with this structure.

Figure 14   250 X   Figure 15   250 X
Alloy IV 9   As Cast   Alloy IV 9   As Rolled

The structure shown in Figure 15 had good ductility at ambient temperature but was
surprisingly brittle at low temperatures. This lack of ductility was probably caused
by a brittle matrix as the β phase in Figure 14 appears to be free from brittle pre-
cipitates. The small black particles in Figure 14 were not caused by hexachlorobenzene
and are too large for nucleant grain refinement.

Figure 16   250 X   Figure 17   250 X
Alloy V 6   As Cast   Alloy V 6   As Rolled

Alloy V 6 was rolled at 800°F and although the structure shown in Figure 17 resulted in good properties, the size and distribution of the β phase is not uniform. Subsequent research indicated that much better properties could have been obtained by rolling this alloy at a much lower temperature.

Figure 18   250 X   Figure 19   250 X
Alloy V 7   As Cast   Alloy V 7   As Rolled

The structure in Figure 19 indicates that this alloy, like alloy V 6, should have been rolled at a lower temperature. The wide spacing between the needle-like β phase is too large to obtain good mechanical properties. The large areas of β were almost unaffected by rolling at 800°F further indicating that a lower rolling temperature could have been advantageous.

| Figure 20 | 250 X | Figure 21 | 250 X |
| Alloy V 8 | As Cast | Alloy V 8 | As Rolled |

Intermetallic compounds have enveloped the β phase in Figure 21 making cross slip between phases difficult. Further development of this alloy does not seem warranted.

The structures of the matrix V alloys are shown in Figures 16 through 21 and are characterized by larger amounts of β phase in the as-rolled condition indicating why, in general, these alloys are a little more ductile than the matrix IV alloys. Some coring can be seen in the as-cast structure of alloy V 6 in Figure 16 and a continuous precipitate is present in the grain boundaries in the as-rolled condition. This alloy (Mg - 2 Th - 2 Ag - 3 Al - 9 Li - 2 Zn - 0.25 Mn) was the most promising of the preliminary alloys. The structure in Figure 17 indicates that much better properties could have been obtained by rolling this alloy at a much lower temperature. The size and distribution of the β phase could have been more uniform had the rolling temperature been lower and the continuous precipitate in the grain boundaries might have been eliminated. Subsequent research on the three alloys ultimately selected for scale-up to 50-pound pilot melts further indicate that alloy V 6 should have been rolled in the 450°F to 500°F temperature range.

Coring is also present in the as-cast structure of alloy V 7, shown in Figure 18, with a great deal of intermetallic compound precipitation in the β phase. Alloy V 7 and IV 9 are similar in composition, with the latter alloy having more zinc and the former containing manganese. Evidently the manganese promotes precipitation in the β phase, a function contrary to its role as a solid solution hardener. The as-rolled structure shows β phase strung out in both directions of rolling and precipitation in the β phase somewhat reduced but still present in quantity. The relatively large spacing between the β phases probably accounts for the mediocre ductility of this alloy; a lower rolling temperature would have increased its ductility and probably the strength as well. The as-cast structure of alloy V 8 shows no coring and elongated β phase. The as-rolled structure in Figure 21 shows the β phase elongated in both directions of rolling and very coarse intermetallic compounds precipitated in the matrix. Intermetallic compounds have also enveloped the β phase making cross slip between phases difficult. Attempts to strengthen this alloy further are expected to meet diminishing returns on time invested.

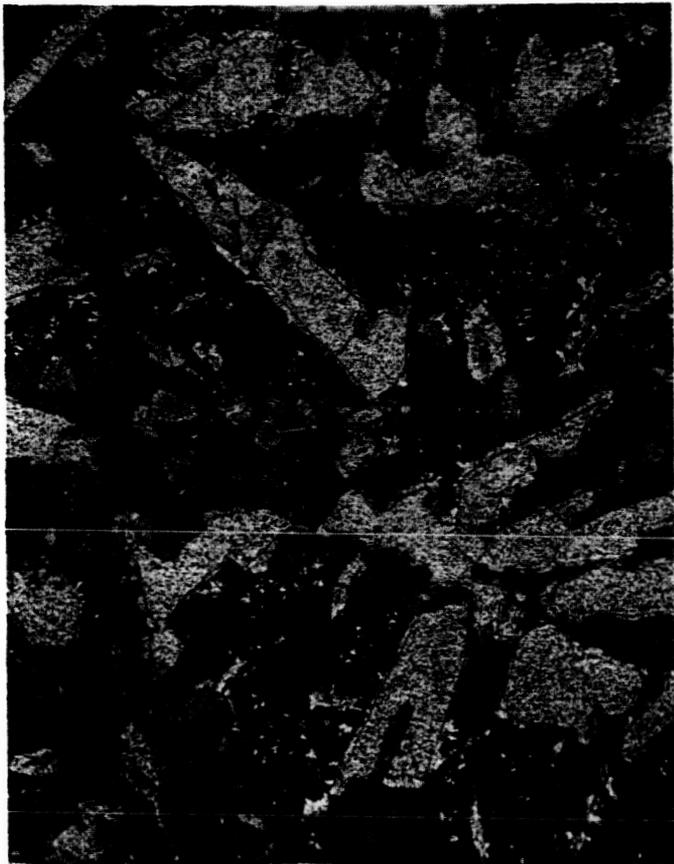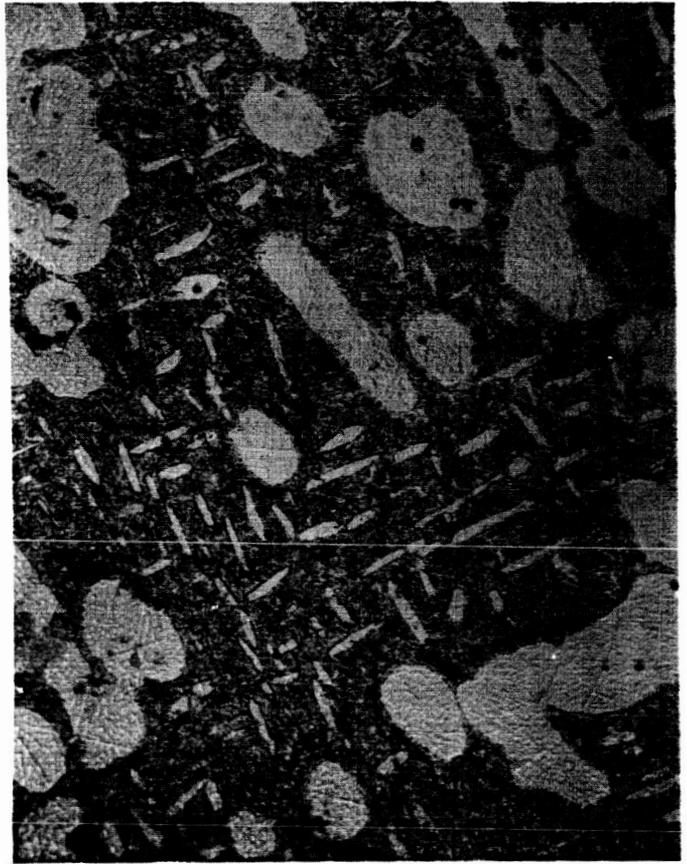The results obtained by testing the matrix IV and matrix V alloys are summarized in Table I. Several of the matrix IV and V alloy specimens were saved for testing at the end of the program so that they could be heat treated similarly to the optimum heat treatment developed for the pilot ingot material. This was done in order to obtain a direct comparison of mechanical properties. A discussion of these results can be found in Section 2.3.3, "Discussion of the Preliminary Test Results".

2.3.2 Results Obtained by Modifying the Composition of Alloy II 4

The five modified II 4 alloys were rolled 75% and 95% into wrought sheet for tensile and heat treating studies. Each alloy was rolled at 300, 400, and 500°F without difficulty. The castings which were rolled at 300°F appeared

TABLE 1

PROPERTIES OF THE PRELIMINARY ALLOYS

Alloy	Rolling Temp. °F	S.H.T. Temp. °F	S.H.T. Time Hrs.	Aging Temp. °F	Aging Time Hrs.	Hardness Re	U.T.S. psi	0.2% offset psi	El. % in 2"	Testing Temp. °F	Remarks
IV 2	800					75	34,200	-	0	68	Cross Rolled
IV 2	800					76	33,300	-	0	68	Cross Rolled
IV 2	800	600	3	200	20	73	32,000	-	0	68	Cross Rolled
IV 2	800	600	3	200	20	70	28,000	26,000	5	68	Unidirectional
IV 2	800	600	3	200	20	72	34,500	28,500	15	68	Unidirectional
IV 2	800	600	3	200	20	70	27,000	25,000	16.3	68	Unidirectional
IV 3	800	800	1	200	1	73	32,200	29,500	17	68	Cross Rolled
IV 3	800	600	3	200	20	74	32,800	27,000	12	68	Cross Rolled
IV 7	800					70	30,600	25,600	30.6	68	Cross Rolled
IV 7	800					85	39,300	35,000	8.5	-320	Cross Rolled
IV 7	800	500	1			81	36,600	31,100	15	68	Cross Rolled
IV 7	800	600	3	200	20	73	31,300	27,000	15	68	Cross Rolled
IV 9	800					84	30,800	25,600	22.8	68	Unidirectional
IV 9	800					83	30,800	25,600	5.5	68	Cross Rolled
IV 9	800					84	41,000	38,700	1.0	-320	Cross Rolled
IV 9	800					82	32,200	30,500	17.1	68	Cross Rolled
IV 9	800					83	32,700	-	0	-320	Cross Rolled
IV 9	800	550	1			84	36,700	34,400	7.0	68	Unidirectional
IV 9	800	800	1	200	1	85	34,800	-	0	68	Unidirectional
IV 9	800	600	3	200	20	78	31,800	26,600	21	68	Unidirectional
IV 9	800	600	3	200	20	77	27,500	-	1	68	Unidirectional
IV 9	800	600	3	200	20	78	30,900	28,000	5	68	Unidirectional
V 6	800					75	26,500	22,200	48.9	68	Cross Rolled
V 6	800	550	1			85	33,300	32,200	11.4	68	Cross Rolled
V 6	800	800	1			93	53,000	-	1	68	Cross Rolled
V 6	800	800	1			92	52,500	-	1	68	Cross Rolled
V 6	800	800	1	200	1	75	42,300	41,300	10	68	Cross Rolled
V 6	800	600	3	200	20	76	38,500	32,500	22	68	Cross Rolled
V 7	800					75	31,400	28,800	10.3	68	Cross Rolled
V 7	800	600	3	200	20	76	34,500	32,000	8	68	Cross Rolled
V 8	800					75	24,100	23,300	26.6	68	Cross Rolled
V 8	800	550	1			85	32,200	31,900	17.1	68	Cross Rolled
V 8	800	800	1	200	1	80	34,800	33,000	12	68	Cross Rolled
V 8	800	800	1	200	1	76	36,600	35,300	10	68	Cross Rolled
V 8	800	600	3	200	20	75	35,000	29,500	12	68	Cross Rolled

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to be the most ductile and the castings rolled at 500°F required less time at temperature between passes for a crack-free sheet product. None of the alloys was given a homogenization heat treatment because the fine grained as-cast structure had very little coring.

The mechanical properties of these alloys as a function of rolling temperature are shown graphically in Figures 22 through 26 and are summarized in Table 2. In every case the lowest rolling temperature produced the most ductile alloy in the as-rolled condition with percent elongation values of greater than 50% for most of the alloys. Removing the hexachlorobenzene from alloy II 4 C to produce alloy II 4 E reduced the ductility somewhat although this effect could not be fully evaluated because alloy II 4 E received a moderate degree of grain refinement from the chilling effect of the mold. Hexachlorobenzene additions to the melt definitely produced grain refinement and did not have any adverse embrittlement effect as indicated by the good elongation values obtained with these alloys.

Raising the rolling temperature did not produce a softening effect. The strength of alloy II 4 A increased with rolling temperature until at 500°F it compared favorably with the as-rolled properties of the unmodified II 4 alloy (51,050 U.T.S., 44,890 Y.S., 16% El. rolled at 720°F). However, the percent elongation values for this 9 Li - 6 Zn - 5 Cd - 5 Ag alloy dropped drastically from 53% to 3% as the rolling temperature increased. The composition of alloy II 4 A is almost identical to the unmodified II 4 alloy except that the latter has less lithium, 7%, and more silver, 6%. This high temperature embrittling effect appeared repeatedly throughout the program especially when the magnesium-lithium alloys were cooled quickly from a high temperature and not subsequently aged. Tests were performed to determine if this effect was caused by lithium depletion resulting from a surface reaction or a straining effect caused by residual stresses produced by rapid cooling. Lithium depletion has been a serious problem with commercial[1] magnesium-lithium alloys and was a problem in this program until it was solved by using a suitable protective atmosphere during hot working or heat treating. However, the effect of a high-temperature embrittlement persisted even after the problem of lithium depletion was solved. The shape of aging curves, discussed separately for the 50-pound pilot melts, indicate that the embrittling effect may be associated with residual stresses and the aging curves, i.e., plots of Strength vs. Aging Time might actually be superimposed curves of aging effects and stress relieving effects. However, very short time aging (actually stress relieving at 200°F) did not produce softening and the brittleness effect persisted until the specimens were heated for longer periods of time. The effect might also be caused by a residual amount of sodium in the matrix as an impurity, but high purity materials were used for all melts and this possibility has been eliminated.

TABLE 2

SUMMARY OF MECHANICAL PROPERTY DATA FOR THE MODIFIED II4 ALLOYS

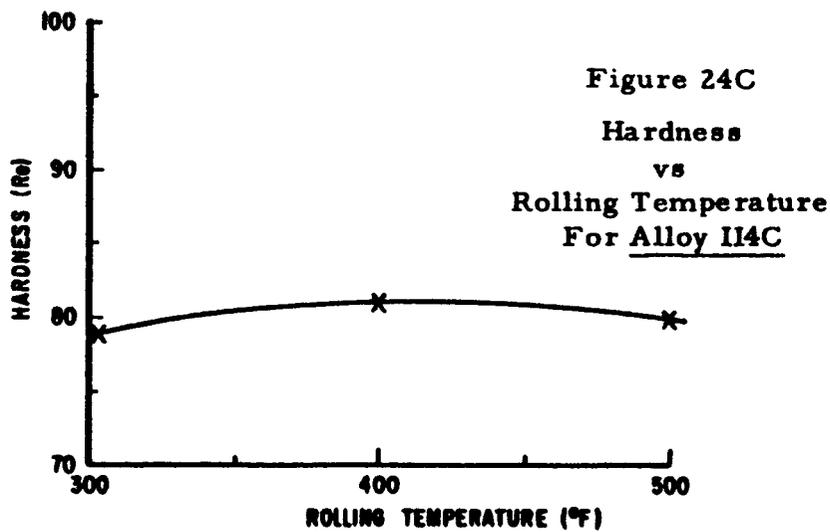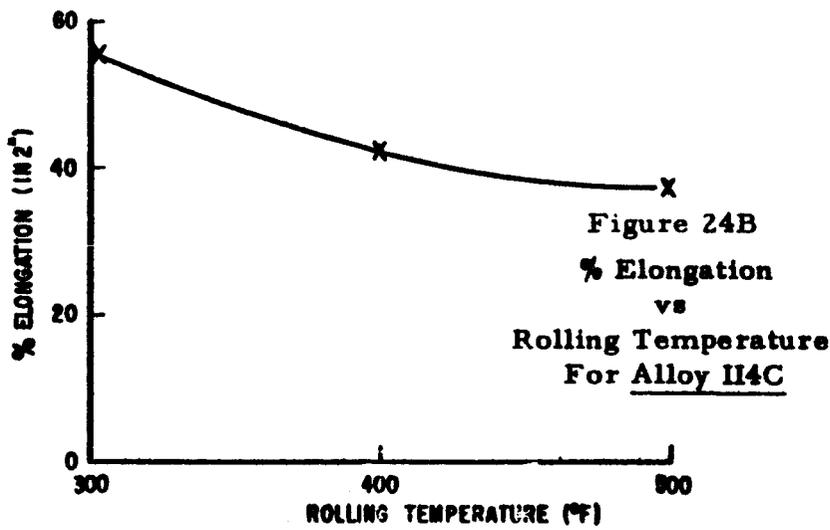| Alloy	U.T.S. psi	0.2% Y.S. psi	El. % in 2"	Hardness Rc	Rolling Temp. °F	S.H.T. Temp. °F	S.H.T. Time Hrs.	Aging Temp. °F	Aging Time Hrs. + Mins.	Testing Temp. °F	Remarks
II 4 A	30,000	26,500	53.0	82	300					68	
II 4 A	38,000	35,000	18.0	88	400					68	
II 4 A	47,300	45,500	3.0	98	500					68	
II 4 A	42,100	-	0.0	98	500	600	1	200	1 + 20	68	
II 4 A	32,000	30,000	30.0	72	400	600	1	200	14	68	
II 4 A	52,500	42,500	9.4	71	400	600	1	200	14	-320	
II 4 A	40,000	33,333	40.6	90	720					68	
II 4 B	32,000	29,000	60	82	300					68	
II 4 B	34,000	32,000	40	86	400					68	
II 4 B	28,000	25,000	30	90	500					68	
II 4 B	53,000	-	-	100	400	600	1	200	½ + 0	68	
II 4 B	53,200	-	1	100	400	600	1	200	½ + 0	68	
II 4 B	48,100	48,000	6.5	98	400	600	1	200	1 + 0	68	
II 4 B	48,200	48,200	7.5	97	400	600	1	200	1 + 0	68	
II 4 B	45,000	43,000	25.0	97	400	600	1	200	1 + 20	68	Data obtained from curve
II 4 B	37,000	35,000	35.0	92	400	600	1	200	2 + 0	68	
II 4 B	38,000	35,200	35.6	91	400	600	1	200	2 + 0	68	
II 4 B	33,700	31,000	36.0	88	400	600	1	200	3 + 0	68	
II 4 B	33,780	31,000	34.0	89	400	600	1	200	3 + 15	68	
II 4 B	29,900	27,000	25.0	85	400	600	1	200	4 + 0	68	
II 4 B	30,100	27,000	27.0	86	400	600	1	200	4 + 0	68	
II 4 B	46,610	-	0.2	100	400	600	1½	200	1 + 0	68	
II 4 B	45,625	45,000	6.2	97	500	600	1	200	1 + 20	68	
II 4 B	46,500	45,500	8.6	96	500	600	1	200	1 + 40	68	
II 4 B	33,000	30,000	36.0		500	600	1	200	3 + 0	68	
II 4 B	50,000	37,500	4.0	86	500	600	1	200	3 + 15	-320	
II 4 C	30,000	28,000	56.0	78	300					68	
II 4 C	31,000	28,000	42.0	81	400					68	
II 4 C	26,500	23,100	37.0	79	500					68	
II 4 C	29,300	24,500	29.5	78	500	600	1	200	1 + 0	68	
II 4 C	27,800	24,800	60.0	60.0	500	600	3	200	3	68	
II 4 C	25,000	22,000	52.0	52.0	500	600	3	200	3	68	
II 4 D	37,000	32,000	37.0	83	300					68	
II 4 D	34,500	32,000	31.0	81	400					68	
II 4 D	32,100	28,000	33	82	500					68	
II 4 D	35,000	29,200	30	83	400	600	1	200	1 + 0	68	
II 4 D	36,000	30,000	29	82	500	600	1	200	1 + 0	68	
II 4 D	43,600	32,900	15.2	65	500	600	1	200	1 + 0	-320	
II 4 D	44,200	33,000	15.0	70.4	400	600	1	200	1 + 0	-320	
II 4 D	59,800	54,500	6.0	79.5	400					-452	As Rolled
II 4 E	28,000	27,000	38	80	400					68	
II 4 E	31,000	29,000	28	82	500					68	
II 4 E	28,700	25,700	35.8	78	300	600	1	200	1 + 20	68	
II 4 E	27,900	23,500	35.5	78	500	600	1	200	1 + 0	68	
II 4 E	43,900	-	0	87	720					68	
II 4 E	25,100	20,700	17.8		720	600	1	200	1 + 0	68	

Figure 22A

Tensile Strength At 68°F
vs
Rolling Temperature
For Alloy II4A

Figure 22B

% Elongation
vs
Rolling Temperature
For Alloy II4A

Figure 22C

Hardness
vs
Rolling Temperature
For Alloy II4A

26

Figure 23A

Tensile Strength At 68°F
vs
Rolling Temperature
For Alloy II4B

Figure 23B

% Elongation
vs
Rolling Temperature
For Alloy II4B

Figure 23C

Hardness
vs
Rolling Temperature
For Alloy II4B

27

Figure 24A

Tensile Strength
vs
Rolling Temperature
For Alloy II4C

Figure 24B

% Elongation
vs
Rolling Temperature
For Alloy II4C

Figure 24C

Hardness
vs
Rolling Temperature
For Alloy II4C

28

Figure 25A

Tensile Strength
vs
Rolling Temperature
For Alloy II4D

U.T.S.

Y.S.

Figure 25B

% Elongation
vs
Rolling Temperature
For Alloy II4D

Figure 25C

Hardness
vs
Rolling Temperature
For Alloy II4D

29

Figure 26A
Tensile Strength
vs
Rolling Temperature
For Alloy II4E

Figure 26B
% Elongation
vs
Rolling Temperature
For Alloy II4E

Figure 26C
Hardness
vs
Rolling Temperature
For Alloy II4E

30

The results obtained by testing the modified II 4 alloys indicate that the embrittlement effect is caused by the high temperature formation of brittle lithium-zinc compounds because the effect became more pronounced as both the lithium and the zinc contents were raised. Very precise metallography of specimens taken later in the program from the scaled-up pilot ingot material showed the formation of very fine precipitates in the lithium rich β phase indicating further that an elevated temperature interaction between lithium and zinc is the mechanism for embrittlement of these alloys. That the effect is not solely associated with a wrought product is illustrated in Figure 27 where the hardness of as-cast magnesium alloys increases with increasing temperature.

Ductility can be restored by aging and the initial drop in hardness, and increase in ductility, must be associated with a loss in coherency between these lithium-zinc compounds and the β second phase; aging then proceeds in a normal diffusion controlled manner. From a standpoint of contributing basic knowledge to the development of magnesium-lithium alloys, additional studies should be made of the brittleness of magnesium-lithium alloys in the solution heat treated condition .

2.3.3 Discussion of the Preliminary Test Results

The results obtained by testing the matrix IV, matrix V and the modified II 4 alloys are grouped together for discussion in this section so that comparisons can be made between the strengthening effect of all the alloying elements studied during the course of the program. In some instances references are made to the results obtained by testing the scaled-up, pilot ingot materials. Although these results were obtained later in the program and discussed more fully in the following sections of this report, they are included in this section to complete the evaluation of the preliminary alloys.

Earlier in the program it was felt that manganese additions to the melt did not strengthen the alloys because its solid solution strengthening effect seemed to be completely masked by other strengthening mechanisms. This does not appear to be the case now that all of the results have been tabulated. Alloy IV 2 and V 6 are almost identical except that V 6 has 0.25% Mn in addition to slightly less zinc. If manganese has no effect, alloy IV would be the stronger of the two, but this is not the case. Alloy V 6 is not only stronger but more ductile than alloy IV 2. One of the alloys chosen for scale-up melting, alloy IA 6, also contained manganese. Considering only the other strengthening elements added to this alloy, it should not be as strong as it was determined to be. The conclusion must be made, therefore, that solid solution strengthening by manganese must be beneficial.

Alloys II 4 A and II 4 B are similar to the unmodified II 4 alloy except that the lithium content was increased from 7% in the II 4 alloy to 8% for II 4 A and finally to 9% for II 4 B. The other changes in the alloy are considered to be minor.
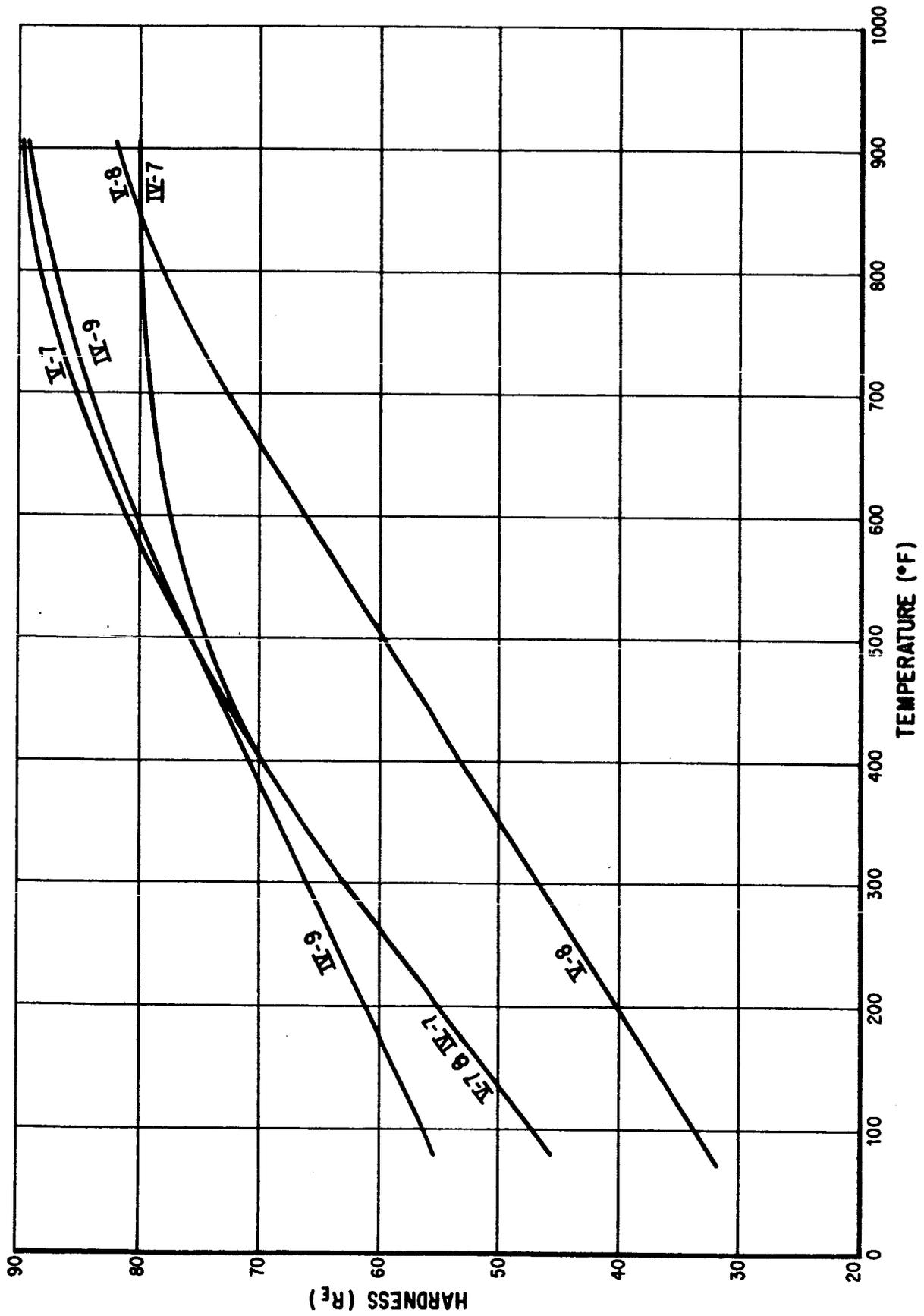
31

Figure 27. Hardness of As-Cast Magnesium Alloys as a Function of Temperature

As expected, the strength decreased and the elongation increased as the percentage of lithium increased.

Alloy IV 3 contained 7% Li with 3% Zn and 6% Al for strengthening. For purposes of discussion we will designate this alloy ZLHA 3726 and compare it with one of the alloys chosen for scale-up, ZLH 972, which has a 45,000 psi tensile strength compared to about 32,000 psi for IV 3. The strengthening effect of aluminum and zinc is not additive in these two phase magnesium-lithium alloys because the effect of 9% Zn is much more pronounced than the effect of 3% Zn plus 6% Al. This is why the statement was made earlier that the strength of alloy IA 6 (Mg - 3 Th - 9 Li - 2 Zn - 4 Ag - 1 Mn - 4 Al) was higher than expected and the strengthening effect of Mn, as well as Ag, must be significant.

Although it may not be as effective as zinc, aluminum does strengthen the alloys. This can be seen by comparing alloy V 6 with the IA 6 pilot ingot material. The two alloys are similar in composition, V 6 (Mg - 2 Th - 9 Li - 2 Zn - 2 Ag - $\frac{1}{4}$ Mn - 3 Al) and IA 6 (Mg - 3 Th - 9 Li - 2 Zn - 4 Ag - 1 Mn - 4 Al), considering that only part of the 1% Mn goes into solution and the effect of silver is not pronounced. The mechanical properties of the two alloys are similar but alloy IA 6 is stronger and more ductile indicating that it was a good selection for scale-up to 50-pound melts.

2.4 Selection of Alloys for the 50-Pound Melts

In spite of the progress made in developing ductility in the magnesium-lithium alloys, none of the preliminary alloys appeared promising enough to be scaled-up directly to 50-pound melts. In order not to neglect the possibility of increasing the mechanical properties of these alloys by a suitable heat treatment, one of the alloys, II 4 B (Mg - 2 Th - 8 Li - 6 Zn - 2.5 Cd), was solution heat treated for one hour at 600°F and aged at 200°F for various times.

The effects of aging time on the mechanical properties of alloy II 4 B are shown in Figures 28 and 29. It is evident that the contractual goals of the program could be met with this alloy but the short aging time, one hour and twenty minutes, indicated that the alloy was not thermally stable and no further development was warranted. According to Clark[2] additions of cadmium to Mg - Li - Zn alloys will stabilize the strength properties because LiCd has the same structure as LiZn but with a larger cell size. Since the unmodified II 4 alloy has more cadmium, 5%, and the modified alloys did not improve upon its mechanical properties, it was decided by mutual consent with the NASA project manager to scale-up the unmodified II 4 alloy to a 50-pound pilot melt.

The stability and elongation values of alloy II 4 D (Mg - 2 Th - 7 Li - 6 Zn - 5 Cd) appeared promising although this alloy, without modifications, did not meet the contractual goals of the program. Correlating the mechanical
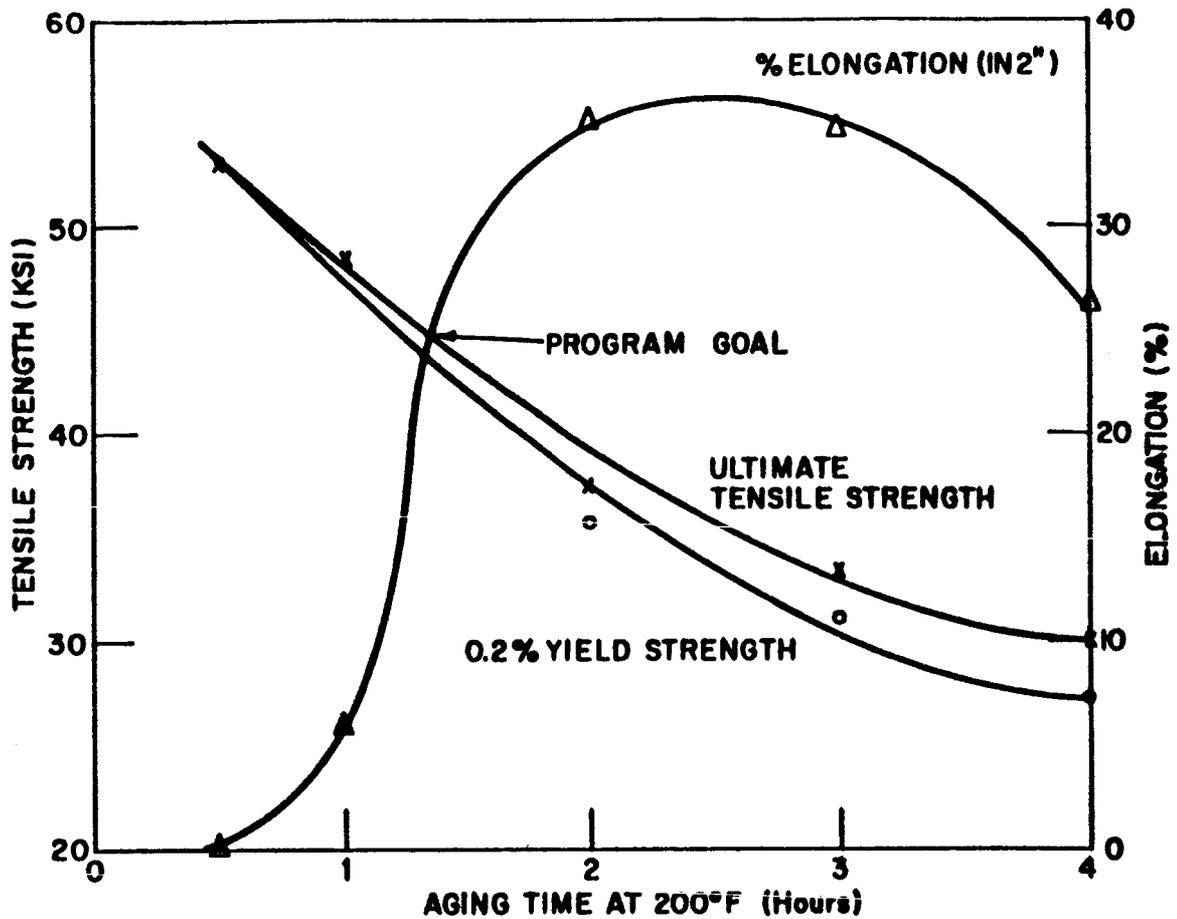
Figure 28. Tensile Strength, Yield Strength, And % Elongation At Ambient Temperature Of Alloy 114B Which Was Rolled Into Sheet At 400°F And Solution Heat Treated For 1 Hour At 600°F.
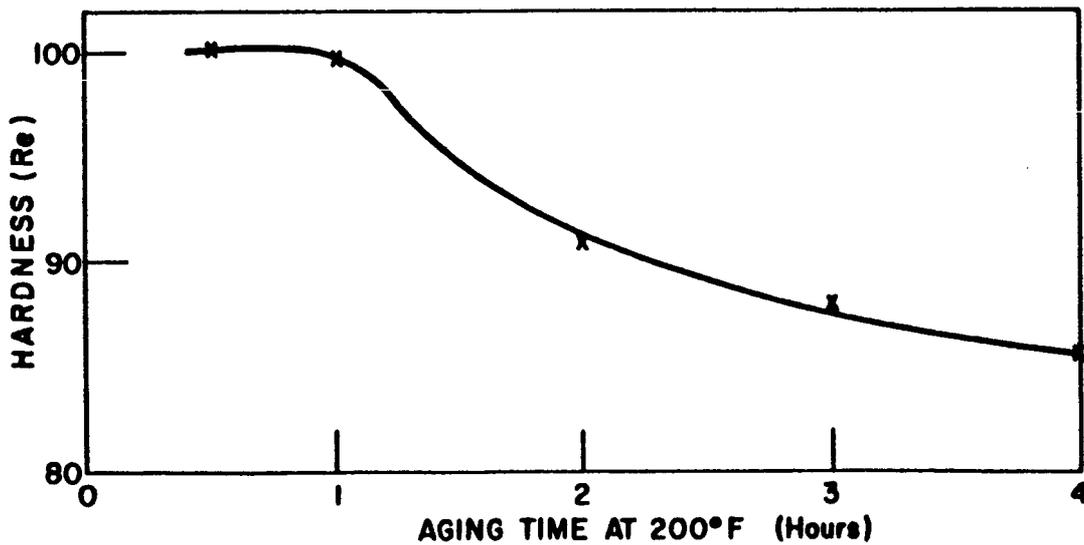

Figure 29. Hardness Vs Aging Time At 200°F For Alloy 114B Which Was Rolled Into Sheet At 400°F And Solution Heat Treated For 1 Hour At 600°F.

34

property data obtained thus far in the program indicated that increasing the zinc content to 9% would greatly increase the ultimate tensile strength and keeping the lithium content at 7% would retain this strength without promoting excessive high temperature embrittling effects. Rather than delaying the scale-up melting program, it was decided to modify alloy II 4 D directly in the 50-pound melts and to omit cadmium from its composition for comparison of thermal stability. The resulting alloy, Mg - 9 Zn - 7 Li - 2 Th , was designated ZLH 972.

The third alloy selected for scale-up to a 50-pound pilot melt was alloy IA 6 (Mg - 3 Th - 9 Li - 2 Zn - 4 Ag - 1 Mn - 4 Al), one of the extra alloys made in the original program. This alloy was originally characterized by poor tensile strength, 25,000 psi, and excellent ductility, 50%, at ambient temperature. At -452°F the tensile strength went up to 66,200 psi with an elongation of 10%. A 50-pound melt would supply enough material for an extensive study program on the effects of thermal-mechanical treatments and it was hoped that such studies would improve the ambient temperature strength of the alloy without a great reduction in ductility.

2.4.1 Preparation of 50-Pound Pilot Melts

While the alloys were being selected for scale-up to 50-pound pilot melts a larger melting facility and permanent mold were being prepared. A sketch of the melting and casting technique used to make the pilot ingots is shown in Figure 30 and a picture of the melting furnace with one of the crucibles in position is shown in Figure 31. A pure magnesium charge was added to the melting crucible which was subsequently sealed and flushed with purified argon before heating. After reaching the melting temperature the alloying elements were added through the alloying port and stirred into the melt by "puddling". As was done during preparation of the preliminary melts, lithium, thorium, zirconium, and manganese were added as master alloys in order to obtain good recovery values. Hexachlorobenzene was added to melts of the ZLH 972 and IA 6 alloys while zirconium was added to alloy II 4 for grain refinement. The grain refiner was always added last just after adding lithium.

While the melt was being charged the temperature of the mold was stabilized at 500°F. Just prior to pouring, the mold was opened and a filter was inserted in the sump. The filter consisted of two concentric screens separated by steel wool for additional filtering action. The wool was lightly packed except at the top and bottom of the screens where it acted as a seal to keep liquid metal from bypassing and getting into the mold cavity without being filtered. Steel wool was also lightly packed into the center of the screens to act as a cushion for the flowing metal in order to eliminate splashing and resulting oxide formation.

RECORDING T-C LEADS

ALLOYING PORT

PUDDLER

SHANK

POURING SPOUT

TO CRANE

MELTING CRUCIBLE

MOLD

GAS FIRED
MOLD HEATER

SAFETY BOX

ARGON

TITANIUM CHIP
GETTERING FURNACE

MICROSIEVE DRYER

RELAY &
INSTRUMENT
PANEL

TILTING MECHANISM

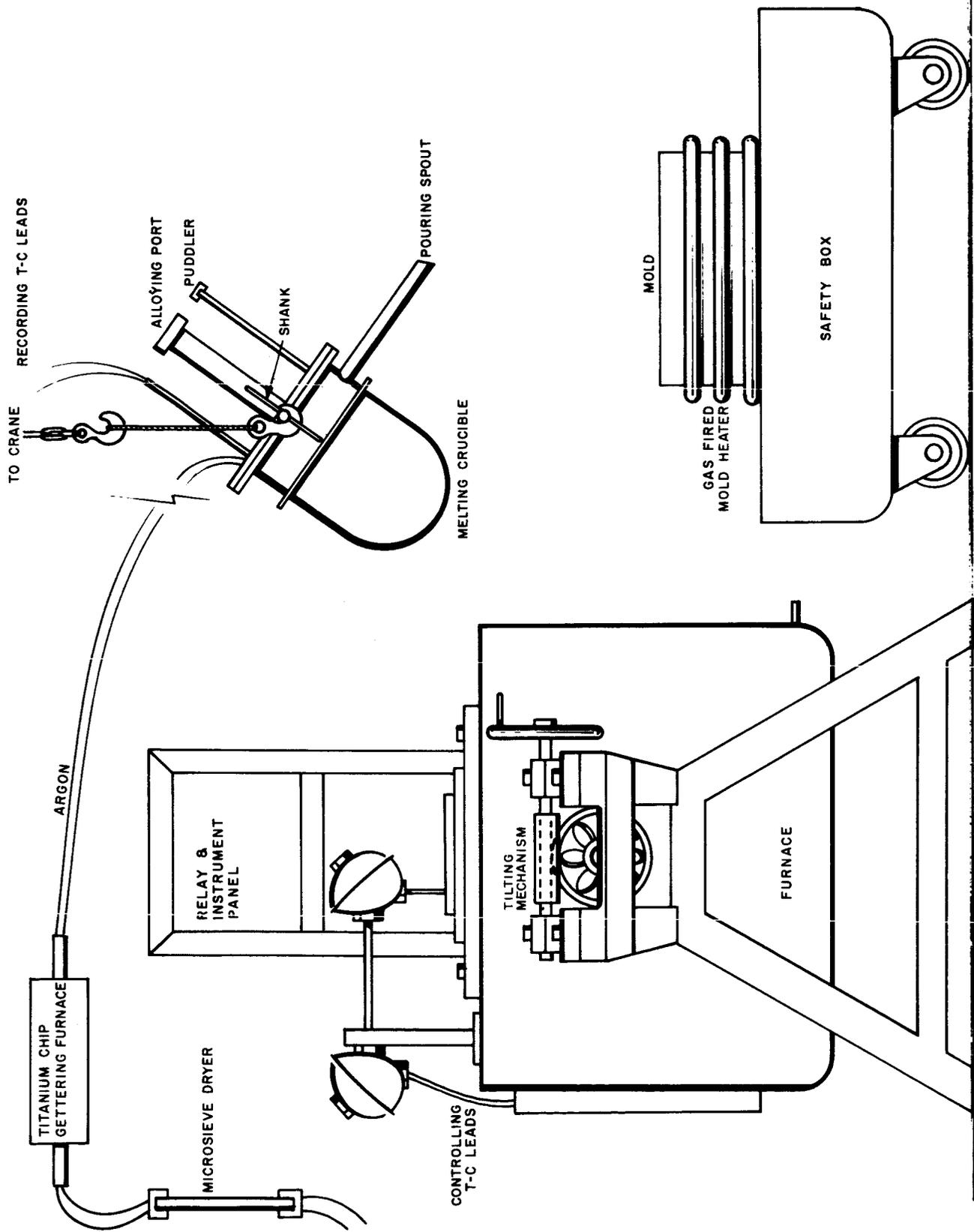CONTROLLING
T-C LEADS

FURNACE

Figure 30 Artist's Conception of Scale-Up Foundry Facility
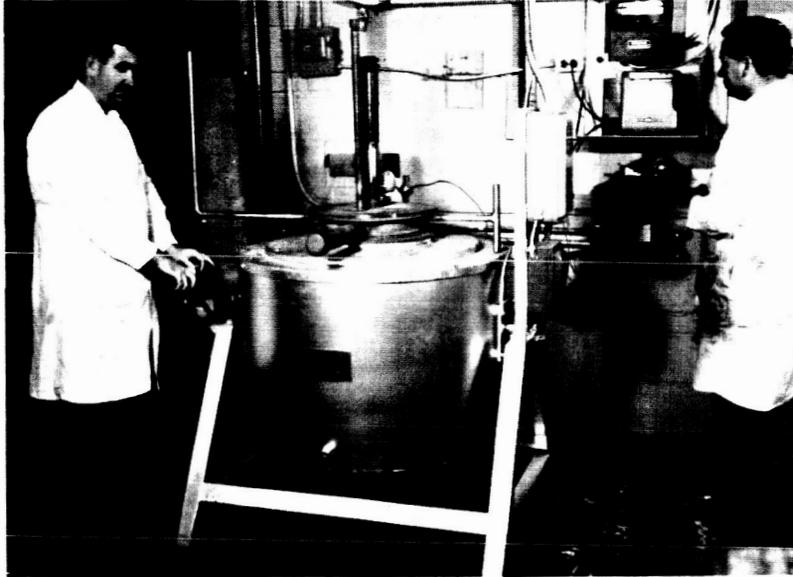
36

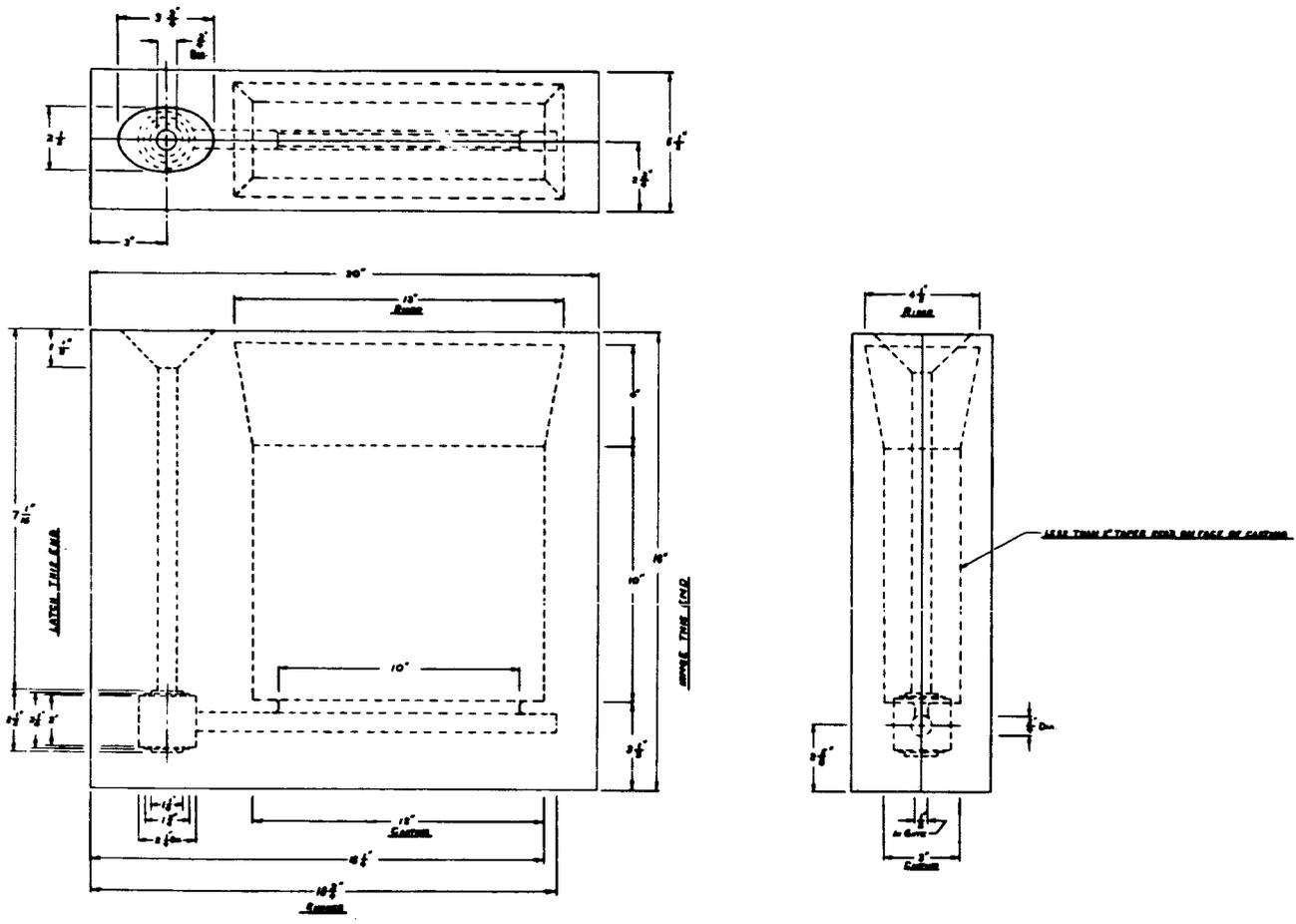Figure 31. Melting Facility For The 50 Pound Magnesium Pilot Melts

After making final preparations and recording the melt temperature and the surface temperature of the mold, the melting crucible was lifted from the furnace by an overhead crane and lowered to the height of the mold where the cap was removed from the pouring spout. Removing the cap allowed argon to escape ahead of the flowing liquid metal so that it could act as a protective blanket and, at the same time, flush the mold ahead of the teemed metal.

After making the first two castings the pouring spout was shortened considerably and reduced in diameter from $2\frac{1}{2}$" to 1" to avoid spillage. After subsequent melts, the mold gating was enlarged slightly. Following good foundry practice, the mold was machined to minimum dimensions, as shown in Figure 32, and metal removed only when found necessary by trial melts and casting. A picture of the nodular iron, permanent mold is shown in Figure 33.

Castings were made of all three alloys, one of which is shown in Figure 34 with the mold coating still adhering to the surface. The runners were fractured from these castings and the surface inspected for dross. In general, the runners were clean, but some isolated particles were visible. In order to insure clean, dross-free material for the testing program, the mold design was altered and additional castings were made of all three alloys. The final pouring operation was made with an enlarged sprue and sump with enough extra screening added to insure that the metal would be choked in the sprue.

2.4.2 Processing the 50-Pound Pilot Melts

The initial castings were rolled at 450°F, 500°F, and 750°F with most of the material rolled at the lowest temperature. Alloy IA 6 rolled so well it was decided to reduce the rolling temperature to 260°F. Preliminary tests indicated that this procedure resulted in improved mechanical properties so most of the IA 6 specimens were rolled at 260°F. The rolling temperature could not be reduced below 450°F for alloy II 4 and ZLH 972 without producing cracks. Thirty percent reductions were made between reheating for each alloy. Surface depletion of lithium during rolling was completely eliminated by heating the rolling stock in either a eutectic mixture of 56% KCl - 44% LiCl salt for the 750°F rolls or in an oil bath for rolling at the lower temperatures. Care was taken to keep the salt mixture hot when it was not in use so that it would not absorb moisture. It was discovered early in the program that lithium depletion was promoted whenever the salt absorbed moisture. Heating the specimens in either the salt or the oil bath insured an accurate and uniform temperature. A few specimens of each alloy were rolled to 0.010" foil without difficulty, but almost all of the material was rolled to 0.10 inch sheet. One specimen of each alloy was hammer forged to foil thickness at its lowest rolling temperature, i.e., 450°F for alloys II 4 and ZLH 972 and 260°F for alloy IA 6, to obtain some

Figure 32. Scale-Up Permanent Mold

The design for the new permanent mold is shown above. The sprue-to-runner-to-gate ratio was chosen to choke the molten stream of metal and reduce turbulence to a minimum. The sump is designed to incorporate concentric screens which will prevent dross from entering the mold cavity. The draft in the sides of the mold cavity is to provide for easy removal of the casting. Nodular iron was chosen for the mold material because of its superior machinability, stability and uniform quality.
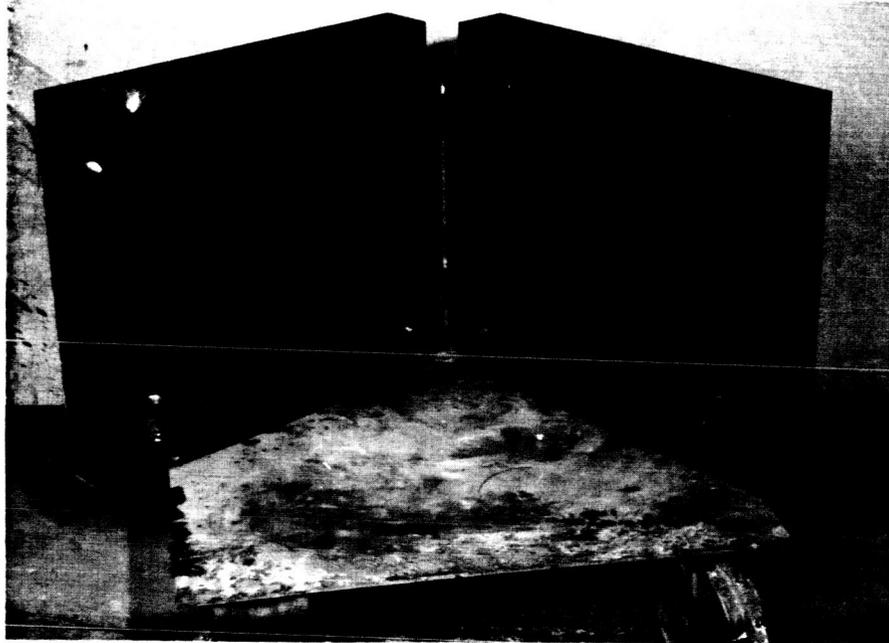
Figure 33. Opened View Of A Nodular Iron Permanent Mold For Producing
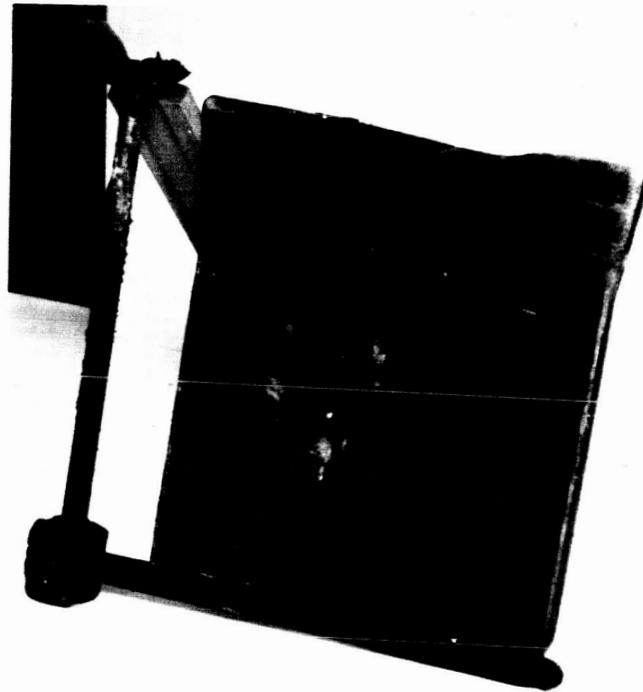Three Inch Thick Ingots Of Magnesium-Lithium Alloys

Figure 34. Casting Made From A 50 Pound Pilot Melt Of Alloy
1A6 (Mg - 3Th - 9Li - 2Zn - 1Mn - 4Al - 4Ag) Showing
The Sprue, Runner, and Gating.

idea of the formability of these materials. Each alloy forged well, especially alloy IA 6, indicating that this alloy is the best choice for difficult-to-form aerospace components.

2.4.3 Metallographic Studies of Wrought Pilot Ingot Material

Studies of the brittle behavior of magnesium-lithium alloys in the T4 condition continued with metallographic examinations of the three alloys chosen for 50-pound pilot melts. Specimens of each alloy which were rolled 90% at 450°F and 750°F were heated for $\frac{1}{2}$ hour and 1 hour at 600°F for these studies. Photomicrographs of the alloys are shown in Figures 35 through 44. Metallographic preparation of these magnesium-lithium specimens was difficult because of surface activity. The procedure used to prepare the specimens was to rough polish them by standard techniques and to final polish them with a Vibromet polisher using Buehler 40-6440AB Magomet polishing compound in an aqueous solution of lithium hydroxide. The etchant was:

ethylene glycol	75 ml
water	25 ml
nitric acid	1 ml

To avoid pitting, metallographic preparation should be continuous up until a good photomicrograph has been obtained.

Photomicrographs of alloy II 4 rolled at successively high temperatures from 450° to 750°F are shown in Figures 35 through 38. Isolated areas of the white β phase appear in the microstructure after rolling at 450° and 500°F while a needle-like β structure appears after rolling at 750°F. It is this needle-like structure that contributed to the excellent ductility of alloy II 4 in the initial program and attempts were made to duplicate it. The structure developed at 750°F is needle-like but too coarse to obtain exceptionally high ductility together with good tensile strength. These photomicrographs indicate that better properties can be obtained by rolling alloy II 4 in the 600° to 700°F temperature range. Heating in this temperature range can promote lithium depletion as it did earlier in the program and a great deal of effort was used to find a suitable oil or neutral salt in which to heat the rolling stock between passes. One salt, an inexpensive nitrite-nitrate mixture called Aeroheat 300, made by the American Cyanamid Co., was found to be inert to magnesium-lithium alloy but it was discovered late in the program and time did not permit tensile specimens to be rolled in this temperature range. The properties obtained from material rolled at 450°F were excellent and it would have been interesting to compare these properties with those obtained after rolling at 650°F.

Photomicrographs of alloy ZLH 972 which was rolled at successively higher temperatures are shown in Figures 39 through 41. The specimen that was rolled at 750°F and solution heat treated for 3 more hours at 600°F shows

Figure 35.   Alloy II 4   100 **X**

This specimen was rolled at 450°F,
solution heat treated for one hour at
600°F, and aged one hour at 200°F.
There is no evidence of aging after
this short aging time.

Figure 36.   Alloy II 4   100X.

This specimen was rolled at 500°F and solution heat treated for ½ hours at 600°F. The photomicrograph shows a two phase α + β structure with the white β phase elongated in the direction of rolling. Very little of the ductile, lithium-rich β phase has segregated to the grain boundaries.

Figure 37.   Alloy II 4   100X

This specimen was rolled at 500°F and solution heat treated
for 1 hour at 600°F. The photomicrograph shows a two phase
α + β structure with the white β phase elongated in the direc-
tion of rolling. Fewer precipitates appear in the β phase after
1 hour at temperature, but enough lithium segregation to greatly
enhance the ductility has not taken place.

Figure 38.   Alloy II 4   100 X

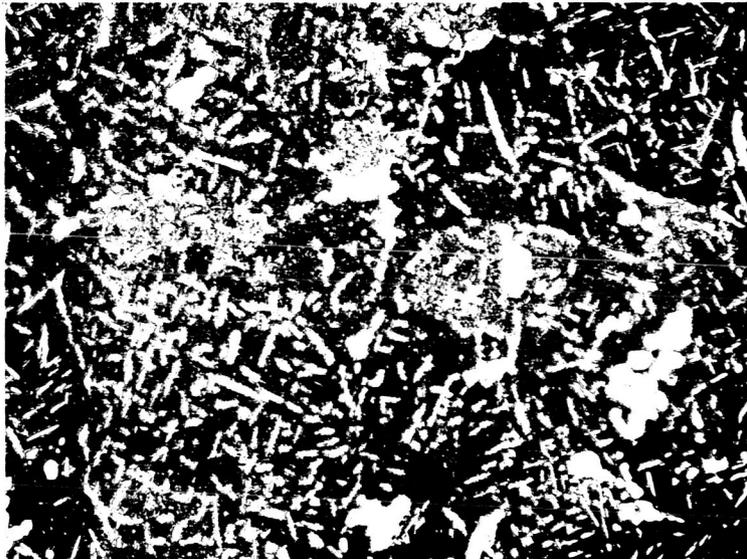This specimen was rolled at 750°F,
solution heat treated for 3 hours at
600°F, and aged 16 hours at 200°F.
The β phase appears as random
needles in the matrix which, in the
past, has resulted in very ductile
material. This structure is undoubt-
edly too coarse for outstanding values
for elongation (16% was obtained) in-
dicating that the optimum rolling
temperature may lie between 450°F
and 750°F.

shows a great deal of precipitation in the β phase which accounts for its brittle behavior in the solution heat treated condition. Rolling at a lower temperature elongated the β phase in the direction of rolling and this phase appears clean and free from precipitates in Figure 39. Solution heat treating this specimen for 3 hours at 600°F, as shown in Figure 40, resulted in the formation of another phase within the β phase which has not been identified and which evidently contributed to the stability of this alloy.

Photomicrographs of alloy IA6 are shown in Figures 42 through 43. Rolling this alloy at 750°F resulted in a material with excellent ductility and poor strength at ambient temperature but with very high strength at -452°F. Rolling at 260°F greatly increased the ambient temperature strength, with a reduction in ductility, and the brittleness in the T4 condition was not as pronounced as in alloys II 4 and ZLH 972 indicating that this brittleness is associated with high lithium together with a high zinc content.

According to Jones and Hogg[3] the T4 brittleness in β magnesium alloys is caused by a continuous network around the grain boundaries which forms when the alloys contain silver or copper. Three of the modified II 4 alloys studied earlier in the program did not contain silver while most of the others did and no correlation was found between brittleness and increased silver content. Clark[2] stated that the cause of embrittlement after solution treatment is obscure and suggests that traces of sodium may be responsible. Very high purity Mg-Li master alloys were used to prepare the melts for this program and the fact that ductility can be recovered makes this hypothesis doubtful. Jones and Hogg could not see precipitation in the β phase after a solution treatment and reported a general coarsening of the grains. An examination of the β phase shown at high magnification in Figure 43 reveals that this coarsening is actually a dispersion of very fine precipitates which are coherent with the β phase. The precipitate is assumed to be $MgLi_2 X$ where X can be either Zn or Al. Aging within the β phase occurs by the rejection of lithium to form MgLi X and LiX with overaging occurring when these compounds lose their coherency with the matrix.

2.5 Mechanical Properties of the Pilot Ingot Material

Standard flat tensile specimens 6 inches long with $2\frac{1}{2}$ inches of reduced area were used in this investigation for test specimens. A concentration factor of $K_t = 10$ was used for the notched specimens which had radii of $0.0015 \pm .0005$ inches at the root of the notch. The welded specimens were made by welding panels and then making tensile specimens from these panels. Welds were made both longitudinal and transverse to the direction of rolling and the specimens machined with the longitudinal welds parallel and the transverse welds perpendicular to the applied load.

Figure 39.   Alloy ZLH 972   100X

This specimen was rolled at 500°F and solution heat treated
½ hour at 600°F.   The white β phase, elongated in the direc-
tion of rolling, appears to contain fewer precipitates than the
β phase in alloy II 4.

Figure 40.   Alloy ZLH 972   100X

This specimen was rolled at 500°F and solution heat treated
for 1 hour at 600°F. This structure appears more equiaxial
than the one shown in Figure 39 and more lithium has segre-
gated to the grain boundaries.

Figure 41. Alloy ZLH972   100 X

This specimen was rolled at 750°F,
solution heat treated for 3 hours at
600°F, air quenched, and was not
superficially aged.   The large β phase
contains a great many precipitates
and an almost continuous network of
the second phase appears in the grain
boundaries.

Figure 42.　　　　Alloy IA 6　　　　100X

This specimen was rolled at 500°F and solution heat treated
for ½ hour at 600°F. The elongated β phase appears rough at
this magnification and a great deal of precipitation can be seen
in the matrix. The time at temperature was not long enough
to dissolve these fine particles.

Figure 43.   Alloy I A 6   2000X

This specimen is the same as the one discussed in Figure 18
but shown here at a much higher magnification.   The rough ap-
pearance of the β phase is caused by very fine precipitates,
assumed to be $MgLi_2Al$.

Figure 44.   Alloy I A 6   100X

This specimen was rolled at 500°F and solution heat treated for 1 hour at 600°F. Fine precipitates have dissolved in the matrix and the β phase appears cleaner.

All specimens were tested in a 20,000 pound capacity Instron testing machine equipped with a continuous load-elongation recorder. A specially constructed cryostat, Cryogenic Inc. Model 307-1, was used for testing at low temperatures. Specimens were tested at ambient temperature, -108°F (dry ice and acetone), -320°F (liquid nitrogen), and -452°F (liquid helium) with the specimens exposed to the cryogen. All specimens were pulled at a rate of 0.1 inch per minute with the strip chart recorder set at 1.0 inch per minute. These speeds gave a magnification factor of 10 which was found convenient for all specimens except the very ductile ones, i.e., those with elongations greater than 30%, where the load-elongation curves were rather long. Calculating the percent elongation values directly from the strip chart gave results which were considered too large and all elongation values tabulated in this report were obtained by marking the specimens prior to testing and measuring the total change in length after the test.

2.5.1 Tensile Testing

The results of short time tensile testing are summarized in Tables 3, 4, and 5. In every case, rolling at the lowest temperature possible without producing edge cracking resulted in the highest ultimate tensile strength. This was especially true for alloy IA 6 where a 260°F roll increased the optimum tensile strength from about 25,000 psi which was obtained earlier in the program, to an average value of about 43,000 psi.

The aging curves in Figure 45 show that the II 4 alloy was unstable after a one-hour solution heat treatment and that a 3-hour solution heat treatment increased its stability considerably. Time did not permit an extensive investigation of the mechanism for this increase in stability but the photomicrograph shown in Figure 40 reveals another phase developing within the unstable β phase which may account for this higher stability. As expected from a longer high temperature heat treatment the grains were more equiaxial and the ends of the elongated β phase were rounded, showing a tendency for this second phase to become spherical. The matrix grain size remained small because of the addition of 2 and 3% thorium for stability.

All three alloys could be air cooled from 600°F to obtain optimum properties, but this rate of quench was marginal and many of these specimens were too soft for testing. In the interest of uniformity all specimens tested at the end of the program were quenched in water resulting in hardness of Re 100 for alloys II 4 and ZLH 972 and about an average of Re 95 for alloy IA 6.

After the solution heat treatment and prior to aging, alloys II 4 and ZLH 972 were hard and brittle whereas alloy IA 6 retained some ductility, approximately 11%. During the first few hours of aging, the strength and hardness of alloys II 4 and ZLH 972 dropped quickly and then became more stable as aging proceeded. The percent elongation of these two alloys increased

Rolling Temp. °F	S.H.T. Temp. °F	S.H.T. Time Hrs.	Aging Temp. °F	Aging Time Hrs.	Hardness Re	U.T.S. psi	0.2% Y.S. psi	El. % in 2"	Testing Temp. °F	Remarks
750						44,300	40,300	18.3	68	As Rolled (average)
750	600	3	200	16	92	38,400	35,400	16	68	
750	600	3	200	16	93	39,800	37,000	12	68	
750	600	3	200	20	90	38,900	34,600	19	68	
750	600	3	200	20	89	37,700	33,400	25	68	
450	800	1	200	1	83	32,800	27,000	16	68	
450	800	1	200	1	84	33,200	28,000	15.8	68	
450					93	47,600	-	0.5	68	As Rolled
450	600	½	200	1	99	51,300	-	0	68	
450	600	1	200	1	99	53,200	50,600	2.7	68	
450	600	1	200	5	89	43,000	36,500	17.0	68	
450	600	1	200	20	84	40,000	34,000	13.7	68	
450	600	2	200	5	90	45,500	39,800	14.0	68	
450	600	2	200	20	89	43,000	34,500	12.0	68	
450	600	3	200	1	97	52,800	49,900	2.0	68	
450	600	3	200	5	93.5	47,600	40,500	15.0	68	
450	600	3	200	20	89	42,500	34,200	16.0	68	
450	600	3	200	20	88	44,000	36,000	16.0	68	
450	600	3	200	20	90	45,500	37,000	14.8	68	
450	600	3	200	20	89	44,200	36,200	14.6	68	
450	600	3	200	20	88	43,600	35,300	15.0	68	
450	600	3	200	20	88	44,200	36,000	15.3	68	
450	600	3	200	20	90	45,600	37,400	15.0	68	
750	600	3	200	20	90	54,500	48,800	2	-320	
450	600	3	200	20	90	53,000	46,300	6.0	-320	
450	600	3	200	20	87	48,000	42,800	7.0	-320	
450	600	3	200	20	88	52,000	46,000	5.3	-320	
450	600	3	200	20	88	51,000	45,000	6.3	-320	
750	600	3	200	20	88	51,000	45,000	6.5	-320	
450	600	3	200	20	89	58,900	51,500	4.0	-452	
450	600	3	200	20	90	60,000	52,200	3.2	-452	
450	600	3	200	20	87	54,000	47,000	5.0	-452	
450	600	3	200	20	87	53,500	47,200	4.3	-452	
750	600	3	200	16	89	61,000	-	1	-452	
750	600	3	200	16	70	56,700	48,200	10	-452	
750	600	3	200	16	89	41,500	-	-	68	Notched/Unnotched = 1.06
750	600	3	200	16	88	46,400	-	-	-452	Notched/Unnotched = 0.78
450	600	3	200	20	88	44,300	-	-	68	Notched/Unnotched = 1.0
450	600	3	200	20	89	41,500	-	-	68	Notched/Unnotched = 0.95
450	600	3	200	20	90	50,300	-	-	-452	Notched/Unnotched = 0.89
750	600	3	200	20	88	40,000	32,000	3	68	Transverse Weld
750	600	3	200	20	78	33,000	28,000	11	68	Transverse Weld
750	600	3	200	20	89	33,600	-	1	68	Longitudinal Weld
450	600	3	200	20	89	47,200	-	1	68	Transverse Weld, 106% Efficient
450	600	3	200	20	88	43,000	37,300	5	68	Transverse Weld, 96.8% Efficient
450	600	3	200	20	87	39,500	34,500	4	68	Longitudinal Weld, 88.8% Efficient
450	600	3	200	20	87	40,000	35,000	5	68	Longitudinal Weld, 90% Efficient

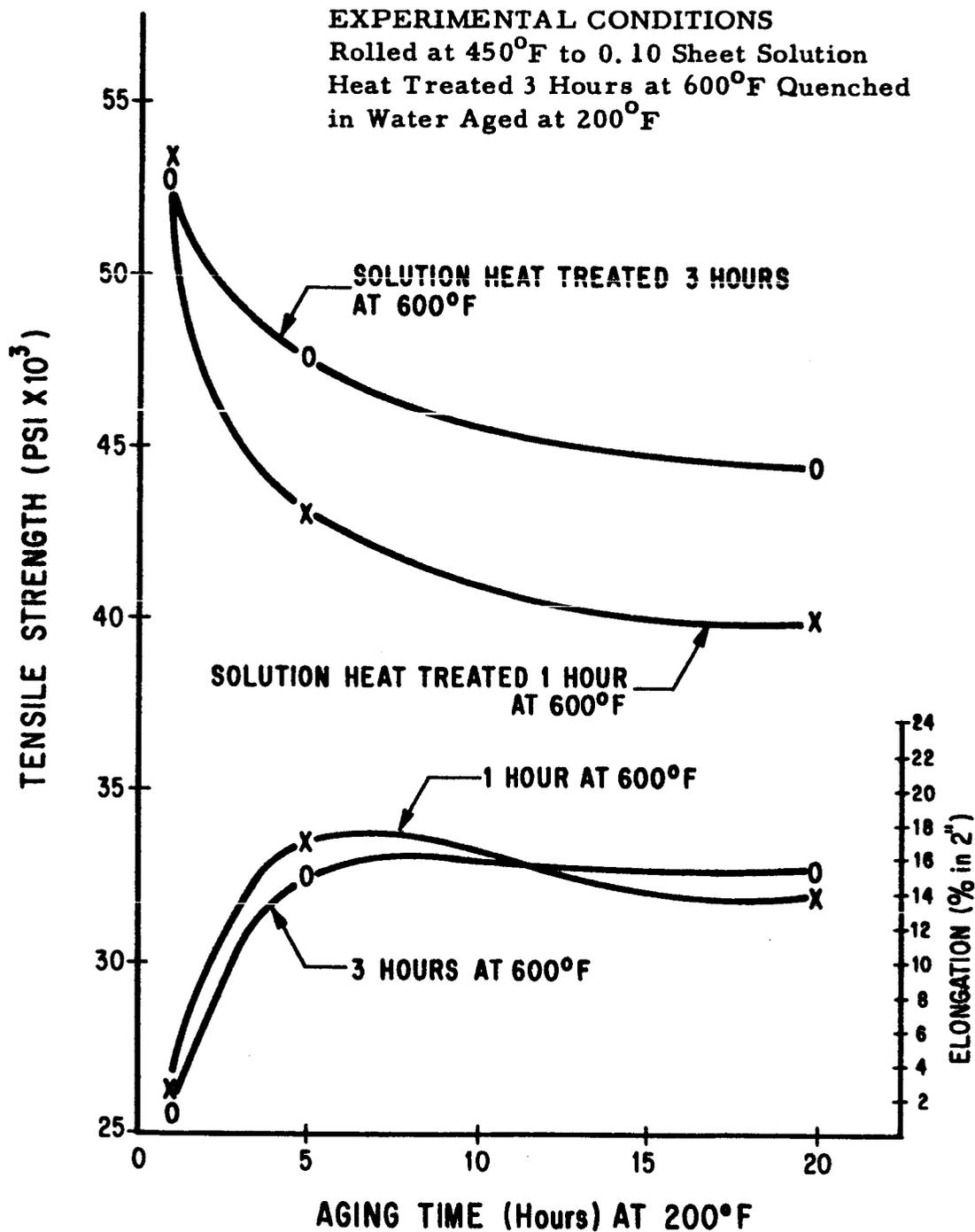The Measured Average Density of Alloy II 4 is 1.82 gm/cc

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Figure 45. The Effect of Aging Time on the Mechanical Properties of Alloy II 4
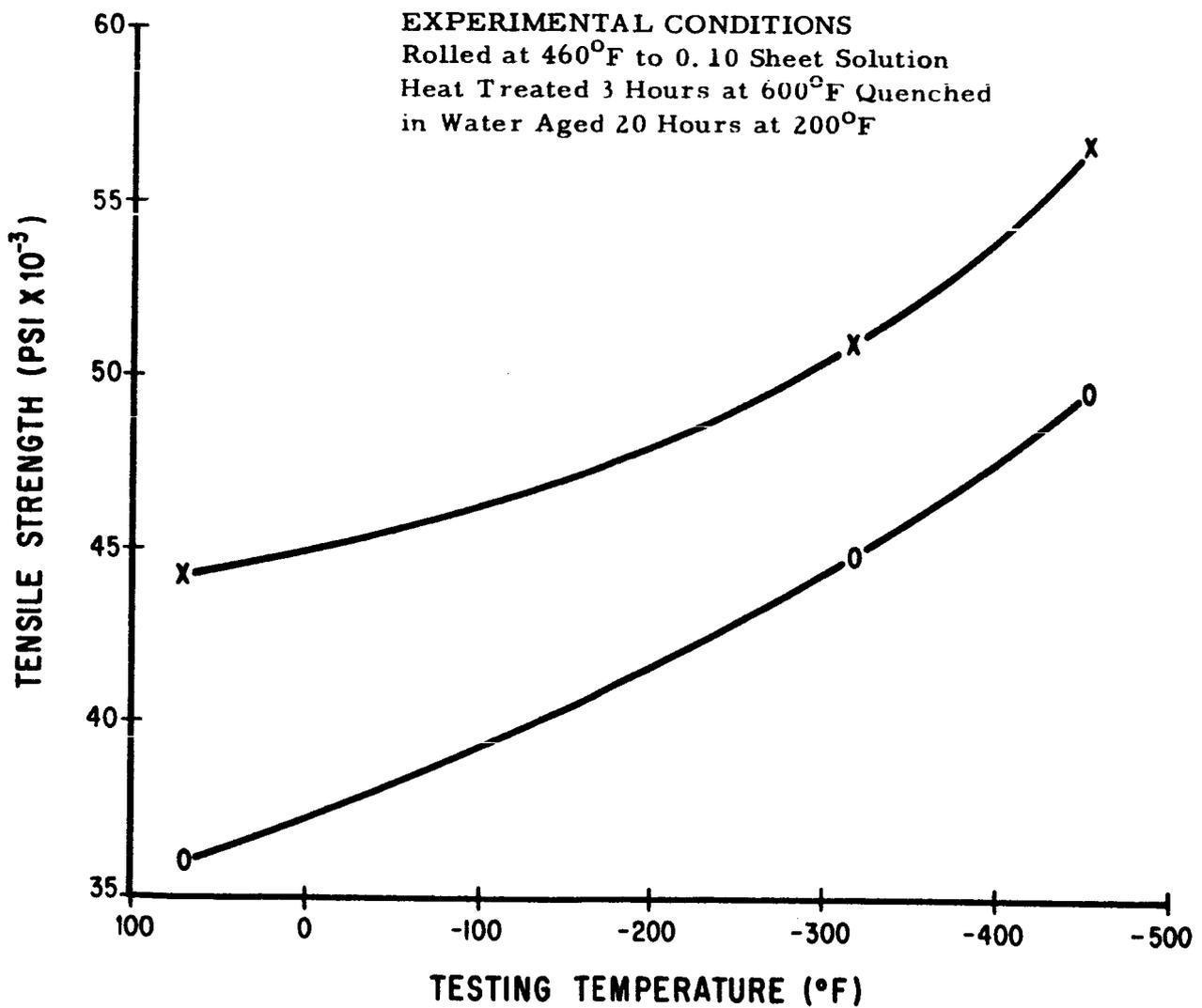
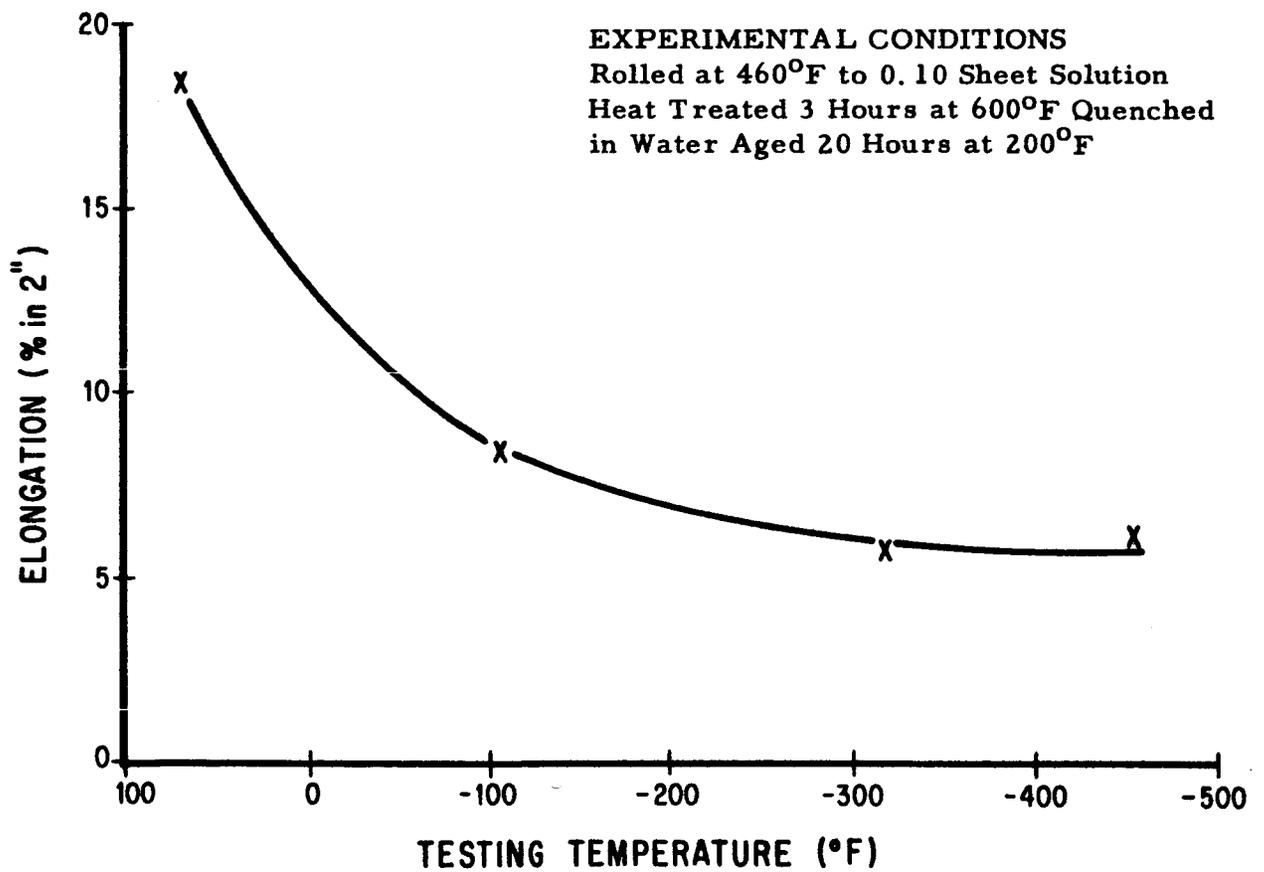Figure 46.   The Effect of Testing Temperature on the Strength of Alloy II 4

Figure 47.   The Effect of Testing Temperature on the Percent
            Elongation (in 2") of Alloy II 4

TABLE 4
RESULTS OF TESTING ALLOY ZLH 972

Rolling Temp. °F	S.H.T. Temp. °F	Time Hrs.	Aging Temp. °F	Time Hrs.	Hardness Rc	U.T.S. psi	0.2% Y.S. psi	El. % in 2"	Testing Temp. °F	Remarks
750	600	3			87	39,700	37,300	16	68	As Rolled
750	600	3	200	16	76	28,000	24,400	35	68	
750	600	3	200	16	79	30,200	28,000	31	68	
750	600	1	200	20	89	30,500	24,400	20	68	
500	600	1	200	2	92	43,200	37,200	5	68	As Rolled
450	600	1	200	5	90	43,000	36,000	12	68	
450	600	1	200	20	86	43,000	36,400	12	68	
450	600	1	200	5	88	43,500	36,000	16	68	
450	600	1½	200	20	88	46,000	45,000	13.5	68	
450	600	3	200	1	78	27,500	23,000	36.6	68	
450	600	3	200	1	98	54,000	45,000	2	68	
450	600	3	200	5	94	50,100	41,300	14	68	
450	600	3	200	20	85	46,000	38,200	17	68	
450	600	3	200	20	89	42,100	37,600	17	68	
450	600	3	200	20	88	40,800	35,400	25	68	
450	600	3	200	20	90	46,500	39,000	16	68	
450	600	3	200	20	90	45,100	37,800	17	68	
450	600	3	200	20	89	47,400	37,000	11.5	-108	
450	600	3	200	20	90	51,100	40,300	9	-108	
450	600	3	200	20	89	49,500	37,800	9	-108	
450	600	3	200	20	88	46,700	36,800	8	-108	
450	600	3	200	20	87	44,800	36,200	5	-108	
450	600	3	200	20	88	51,200	49,200	5.0	-320	
450	600	3	200	20	88	50,100	49,000	6.0	-320	
450	600	3	200	20	89	53,300	48,000	4.0	-320	
450	600	3	200	20	88	50,000	47,900	5.0	-320	
450	600	3	200	20	88	51,400	50,000	4.0	-320	
450	600	3	200	20	89	51,200	49,000	4.8	-320	
450	600	3	200	20	87	57,500	54,000	5	-452	
450	600	3	200	20	88	56,000	52,700	6	-452	
450	600	3	200	20	88	52,000	49,200	6.3	-452	
450	600	3	200	20	90	58,700	51,700	5.8	-452	
450	600	3	200	20	87	50,000	48,700	6	-452	
450	600	3	200	20	90	58,600	50,300	6	-452	
450	600	3	200	20	88	57,500	50,000	8.5	-452	
750	600	3	200	16	90	35,100	-	-	68	Notched/Unnotched .78
750	600	3	300	16	88	48,200	-	-	-452	Notched/Unnotched .89
450	600	3	200	20	88.5	40,200	-	-	68	Notched/Unnotched .895
450	600	3	200	20	89	43,000	-	-	68	Notched/Unnotched .96
450	600	3	200	20	90	50,000	-	-	-452	Notched/Unnotched .896
450	600	3	200	20	87	42,000	37,000	5	68	Transverse Weld, 94.5% Efficient
450	600	3	200	20	88	41,000	26,000	6	68	Longitudinal Weld, 92% Efficient
450	600	3	200	20	88	28,000	26,000	2	68	Transverse Weld, Dross in Weld

The Measured Average Density of Alloy ZLH972 is 1.81 gm/cc

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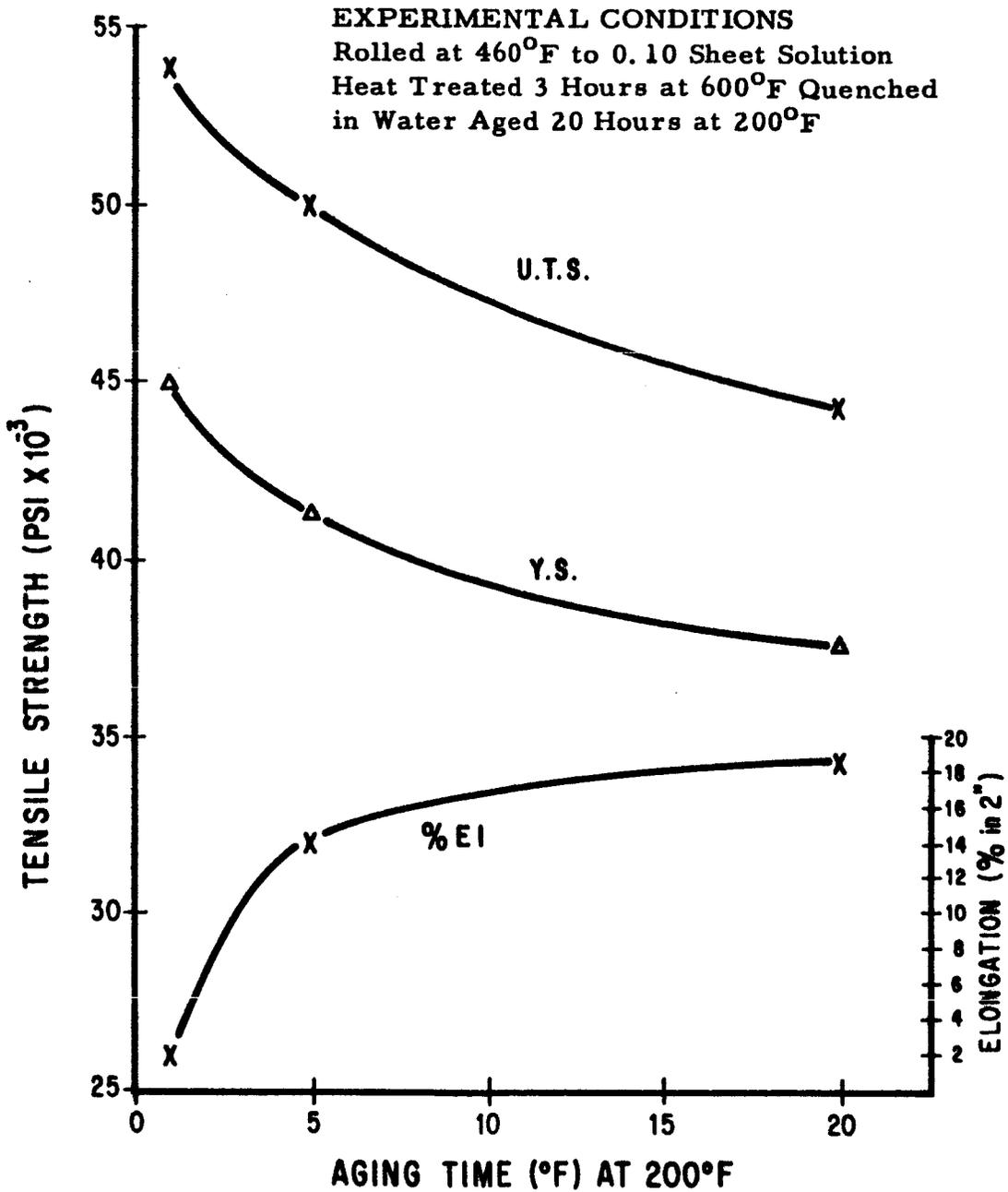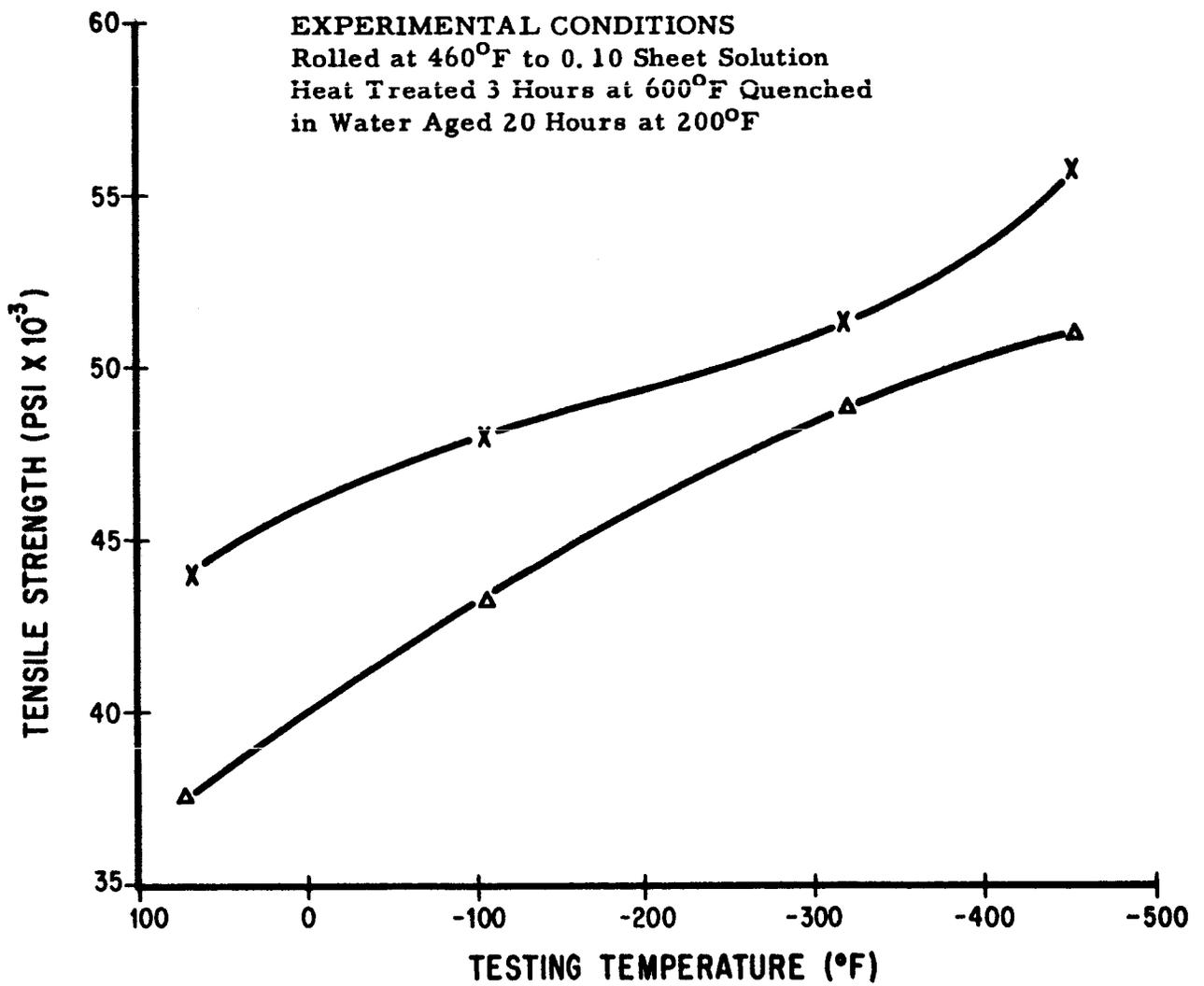Figure 48. The Effect of Aging Time at 200°F on the Mechanical Properties of Alloy ZLH 972

Figure 49.   The Effect of Testing Temperature on the Strength of Alloy ZLH 972

EXPERIMENTAL CONDITIONS
Rolled at 460°F to 0.10 Sheet Solution
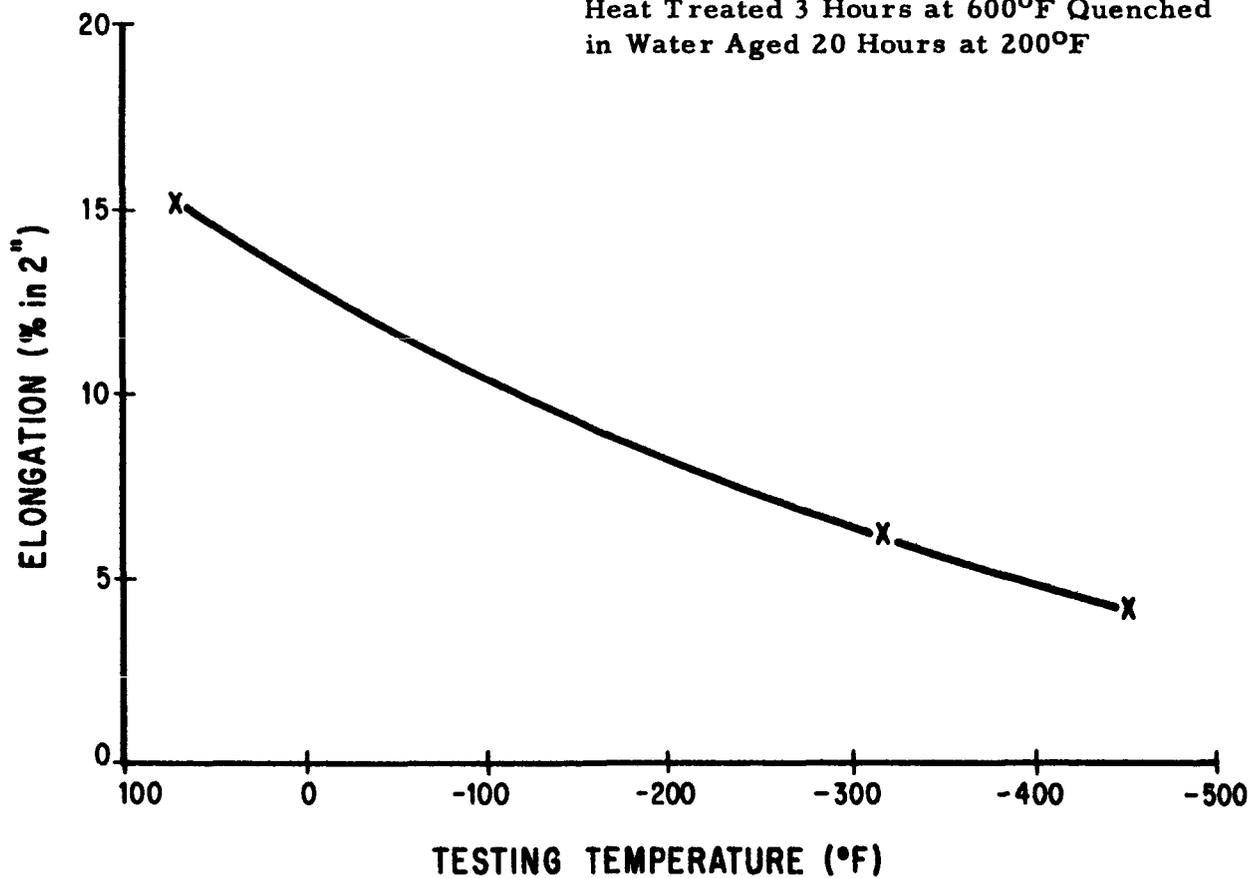Heat Treated 3 Hours at 600°F Quenched
in Water Aged 20 Hours at 200°F

Figure 50.   The Effect of Testing Temperature on the Percent
Elongation (in 2") of Alloy ZLH 972

TABLE 5
RESULTS OF TESTING ALLOY IA6

Rolling Temp. °F	Temp. °F	Time Hrs.	Temp. °F	Time Hrs.	Hardness Rc	U.T.S. psi	0.2% Y.S. psi	El. % in 2"	Testing Temp. °F	Remarks
750					79	28,200	25,000	30.8	68	As Rolled, Coarse Grained
750	850	1	200	½	74	24,700	21,800	50	68	Coarse Grained
750	850	1	200	½	76	25,800	23,000	15	68	Coarse Grained
500	600	1	200	1	78	28,900	25,700	32	68	
500	600	1	200	2	78	26,500	23,200	30.4	68	
260	600				78	34,000	31,500	37	68	As Rolled
260	600	1	200	1	88	40,200	40,000	17	68	
260	600	3	200	1	88	39,500	38,500	19	68	
260	600	3	200	1½	88	40,000	39,000	18	68	
260	600	3	200	2	81	38,000	38,000	15	68	
260	600	3	200	2	84.5	37,300	37,200	17	68	
260	600	3	200	2	90	39,200	39,200	17	68	
260	600	3	200	20	81	36,000	35,000	20	68	
260	600	3	200	½	91	50,600	48,000	11	68	
260	600	3	200	1½	90	49,000	48,000	12.3	68	
260	600	3	200	5	88	46,400	46,000	15.5	68	
260	600	3	200	20	87	43,000	37,800	16	68	
260	600	3	200	20	86	42,500	42,000	16	68	
260	600	3	200	20	86	43,500	43,000	15.5	68	
260	600	3	200	20	85	39,500	39,000	18	68	
260	600	3	200	20	86	40,500	39,500	17	68	
260	600	3	200	20	87	42,700	39,600	20	68	
260	600	3	200	20	89	52,200	46,300	8.2	-108	
260	600	3	200	20	87	46,000	40,100	8.5	-108	
260					85	42,800	36,500	13.0	-108	
260	600	3	200	20	85	44,000	38,400	6.0	-108	
260					88	49,100	40,100	14.0	-108	
260	600	3	200	20	88	49,100	42,100	8.0	-108	
260	600	3	200	20	85	42,600	36,400	13.0	-108	
260	600	3	200	20	86	43,500	37,700	10.5	-108	
450	600	1	200	1	80.5	49,000	42,600	14	-320	
450	600	1	200	2	82	48,100	42,000	14	-320	
260	600	3	200	20	88	58,800	49,800	7	-320	
260	600	3	200	20	88	53,100	47,500	4	-320	
260	600	3	200	20	87	46,700	42,500	-	-320	Broke in Pin Area
260	600	3	200	20	86	49,000	42,600	5.0	-320	
260	600	3	200	20	88	53,500	-	-	-320	Broke in Pin Area
260	600	3	200	20	89	55,300	50,100	7.0	-320	

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260	600	3	200	20	88	60,700	60,000	4	-452	
260	600	3	200	20	90	70,200	66,000	2	-452	
260	600	3	200	20	89	64,300	61,200	3	-452	
260	600	3	200	20	88	64,400	61,200	3	-452	
260	600	3	200	20	88	65,700	-		-452	Broke in Pin Area
260	600	3	200	20	87	61,000	60,000	4	-452	
260	600	3	200	20	86	59,300	50,200	8.5	-452	
260	600	3	200	2	85	38.400	-	-	68	Notched/Unnotched = 0.91
260	600	3	200	20	89	43,600	-	-	68	Notched/Unnotched = 1.03
260	600	3	200	20	80	44,000	-	-	68	Notched/Unnotched = 1.04
260	600	3	200	20	87	58,200	-	-	-452	Notched/Unnotched = 0.91
260	600	3	200	20	88	58,800	-	-	-452	Notched/Unnotched = 0.92
260	600	3	200	20	86	26,000	22,400	12.5	68	Transverse Weld, 61.7% Efficient
260	600	3	200	20	86	27,700	23,300	6.6	68	Transverse Weld, 65.5% Efficient
260	600	3	200	20	88	34,200	29,400	8	68	Transverse Weld, 81% Efficient
260	600	3	200	20	89	33,900	29,200	9	68	Longitudinal Weld, 80.5% Efficient

The Measured Average Density of Alloy I A 6 is 1.57 gm/cc
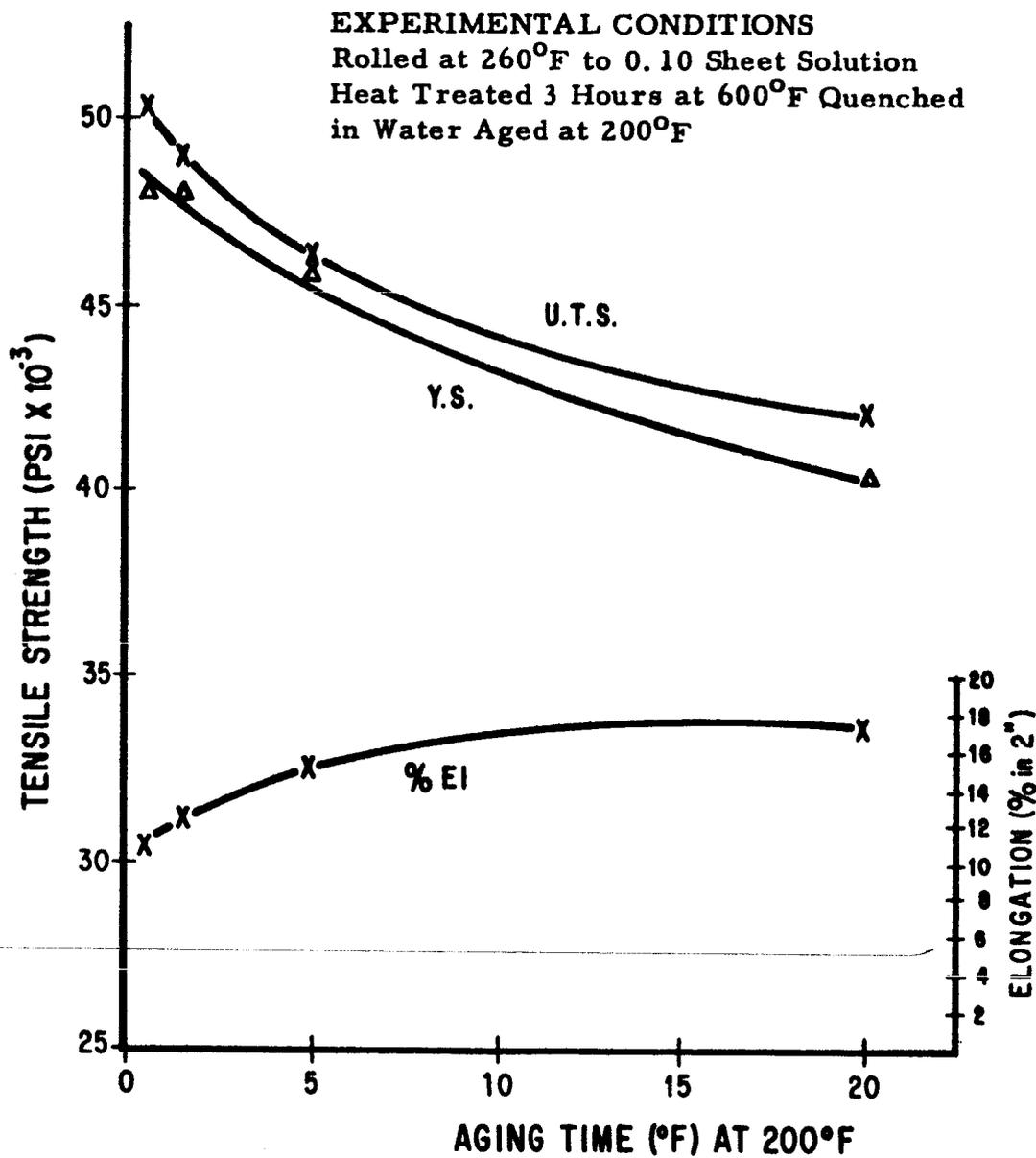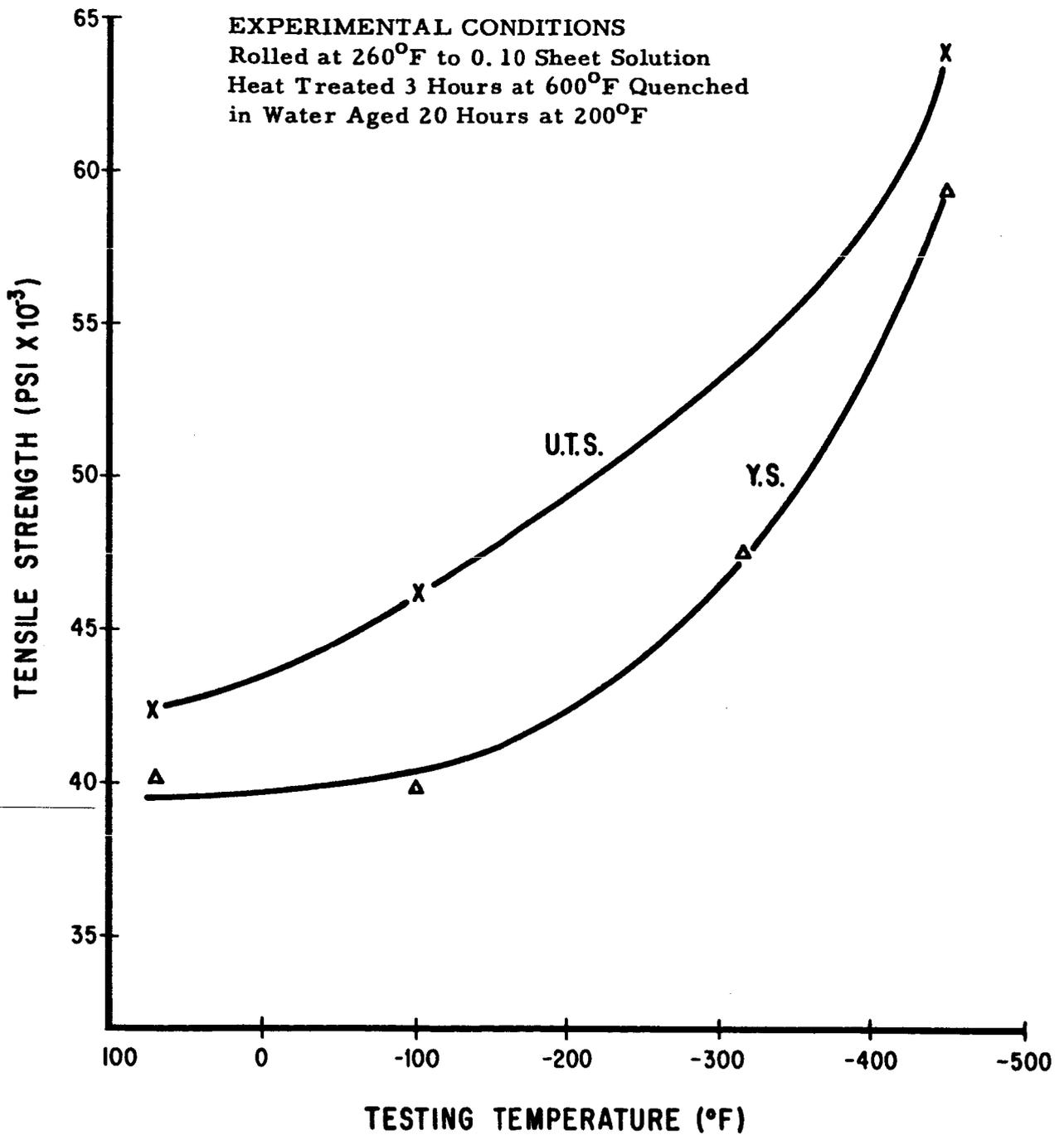
Figure 51. The Effect of Aging Time on the Mechanical Properties of Alloy I A 6

Figure 52. The Effect of Testing Temperature on the Mechanical Properties of Alloy I A 6
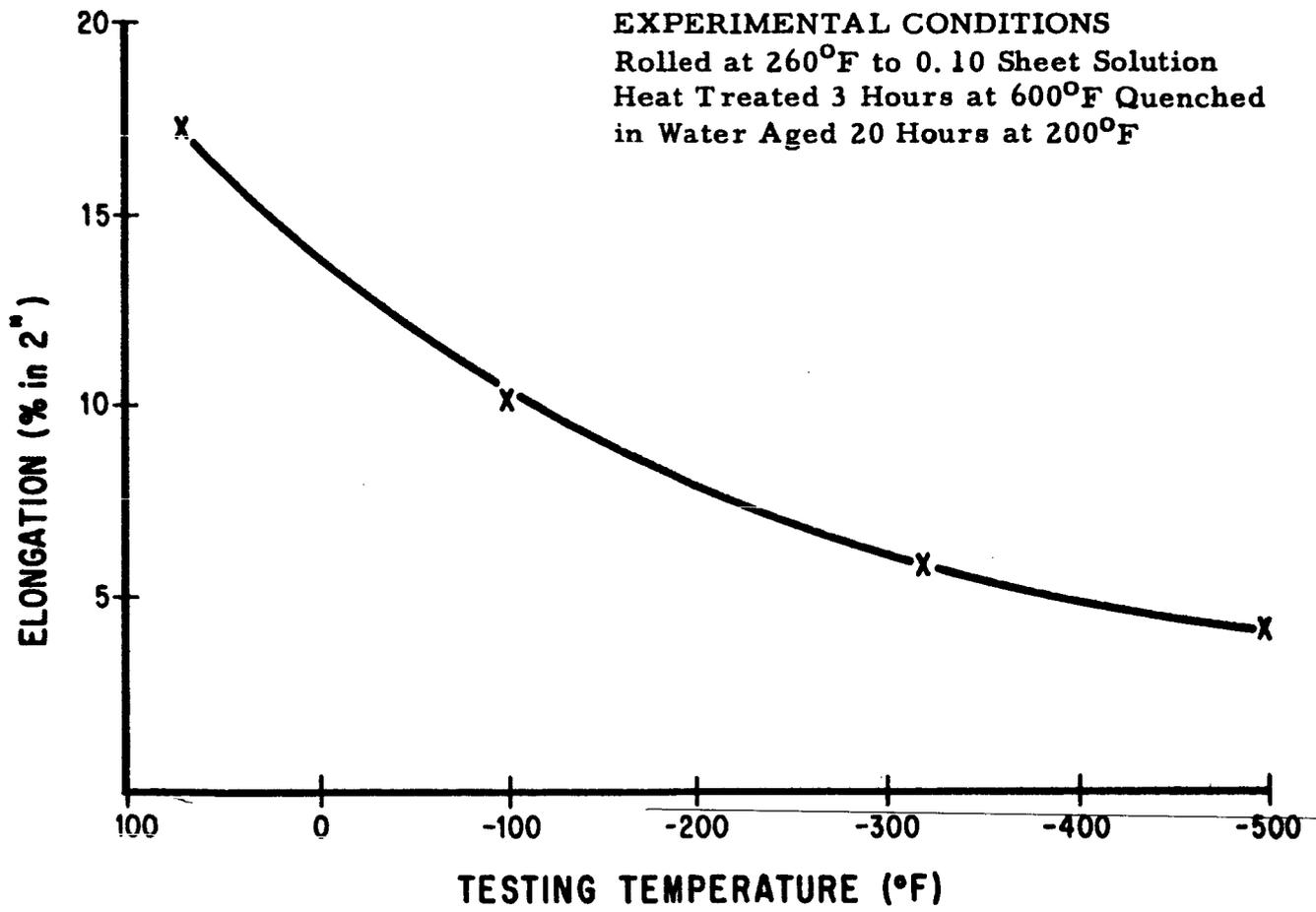
EXPERIMENTAL CONDITIONS
Rolled at 260°F to 0.10 Sheet Solution
Heat Treated 3 Hours at 600°F Quenched
in Water Aged 20 Hours at 200°F

Figure 53. The Effect of Testing Temperature on the Percent
Elongation of Alloy IA 6

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sharply after a few hours of aging while the increase in ductility of alloy
I A 6 was less drastic. As discussed in Sections 2.3.3 and 2.4.3 no correla-
tions could be made between this initial brittleness, and initial change in
mechanical properties after aging, and a trace amount of sodium or electron
concentration of the alloys. Part of the initial drop in strength properties
must be attributed to stress relieving but attempts to determine if the hardness
of the alloys would decrease drastically after very short aging (essentially
stress relieving) and then increase upon further aging failed to change the shape
of the aging curves, indicating that stress relieving is not the entire mechanism.

Alloys II 4 and ZLH 972 have a relatively large amount of lithium and
zinc, close to 1:1 ratio, while alloy I A 6 contains three times as much lithium
as zinc. The other strengthening elements in alloy I A 6 are aluminum, manga-
nese and silver. These results indicate that initial brittleness is caused by a
compound in the lithium-rich β phase, such as $MgLi_2Zn$, which overages very
quickly and that $MgLi_2Al$, or a similar compound, has a less embrittling effect.
The aging curves were not extended long enough to determine which of the alloys
is more stable but this should be done along with x-ray diffraction studies in
order to contribute to a more thorough understanding of the aging mechanism in
these alloys.

For the specimens given the optimum thermal-mechanical treatments
the ambient temperature tensile strength of alloy II 4 varied between 42,500
and 45,600 psi with corresponding elongation values 16 and 15%. Under the
same conditions alloy ZLH 972 varied from 40,800 to 46,500 psi and from 25
to 16% elongation. Alloy I A 6 varied from 39,500 psi to 43,500 psi with the
elongation varying between 20% and 15.5%. Except for the one specimen that
had a low tensile strength of 40,000 psi, alloy ZLH 972 came bery close to meet-
ing the ultimate tensile strength goals of the program. No problem was en-
countered in meeting the yield strength goals, but the average elongation values
for all three alloys were slightly less than the 20% goal. The strength of
each alloy increased with decreasing temperature with alloy I A 6 reaching a
maximum tensile strength of 70,200 psi at -452°F. All three alloys had usable
ductility at -452°F although the goal of 15% was not attainable.

In addition to the ultimate tensile strength, yield strength (.2% offset)
and % elongation (within 2"), the modulus of elasticity was obtained for each
alloy. When the modulus of elasticity was calculated directly from the slope
of the load vs. elongation curves recorded automatically on the strip charts,
values of 7 to 7.2 x 10^6 were obtained at ambient temperature. These values
were considered to be too high and they were not listed in Tables 3, 4 or 5.
Instead, corrections were made for relaxation effects and these new values
ranged from 6.5 x 10^6 at ambient temperature to 8.2 x 10^6 at -452°F. The
method used for calculating the elastic modulus and the very slight changes in
the slope of the curves did not permit a comparison to be made between the
elastic modulus of each alloy.

2.5.2 Notched Tensile Strength

Notched tensile specimens ($K_t = 10$) were tested at ambient temperature and at $-452^\circ F$. The alloys developed in the initial 12-month program were notch sensitive, but with the longer aging times developed for the alloys in the current program the specimens became less sensitive to notches. All specimens were close to a notched/unnotched ratio of 1.0 at ambient temperature and 0.9 at $-452^\circ F$. The average ultimate tensile strengths were used for the unnotched strength values which accounts for the variations obtained for notched sensitivity.

2.5.3 Weldability of the Pilot Ingot Material

For most magnesium alloys an increase in alloy content increases the solidification range but lowers the shrinkage and the solidus and liquidus temperatures. For alloys containing both aluminum and zinc, the aluminum content aids weldability and zinc induces hot shortness and resulting weld cracking. Thorium, which provides strength at elevated temperatures, greatly enhances weldability.

All three alloys developed in this program were easy to weld using the TIG method with an a-c machine that contained a high frequency arc stabilizer. A picture of a typical welded panel is shown in Figure 54. All weld beads were removed from the surface by machining. The specimens tested and recorded in Tables 3, 4 and 5 were welded by using filler rods made of the parent material. A few specimens were welded using other weld rod material but the welds were poor and were not included in the testing program. At the beginning of the program considerable trouble was encountered in obtaining dross-free welds but this problem was solved by using an excess amount of cover gas and by cleaning the filler rods just prior to welding.

Although none of the specimens developed cracks, the weld joint efficiency of alloy IA 6 was the poorest of the three alloys under development while alloy ZLH 972 was easy to weld and had good efficiency. These results are surprising in view of the high zinc content of alloy ZLH 972 and the fact that alloy IA 6 was the only one that contained aluminum. All three alloys contained a small amount of thorium which may have aided in preventing crack formation. Since all three alloys were welded with parent metal filler rods and stress relieved during aging, stress-corrosion cracking is expected to be negligible.

2.6 Coatings Feasibility

A requirement for the alloys under development in this program is that they must be coatable by standard means although development of a new coating
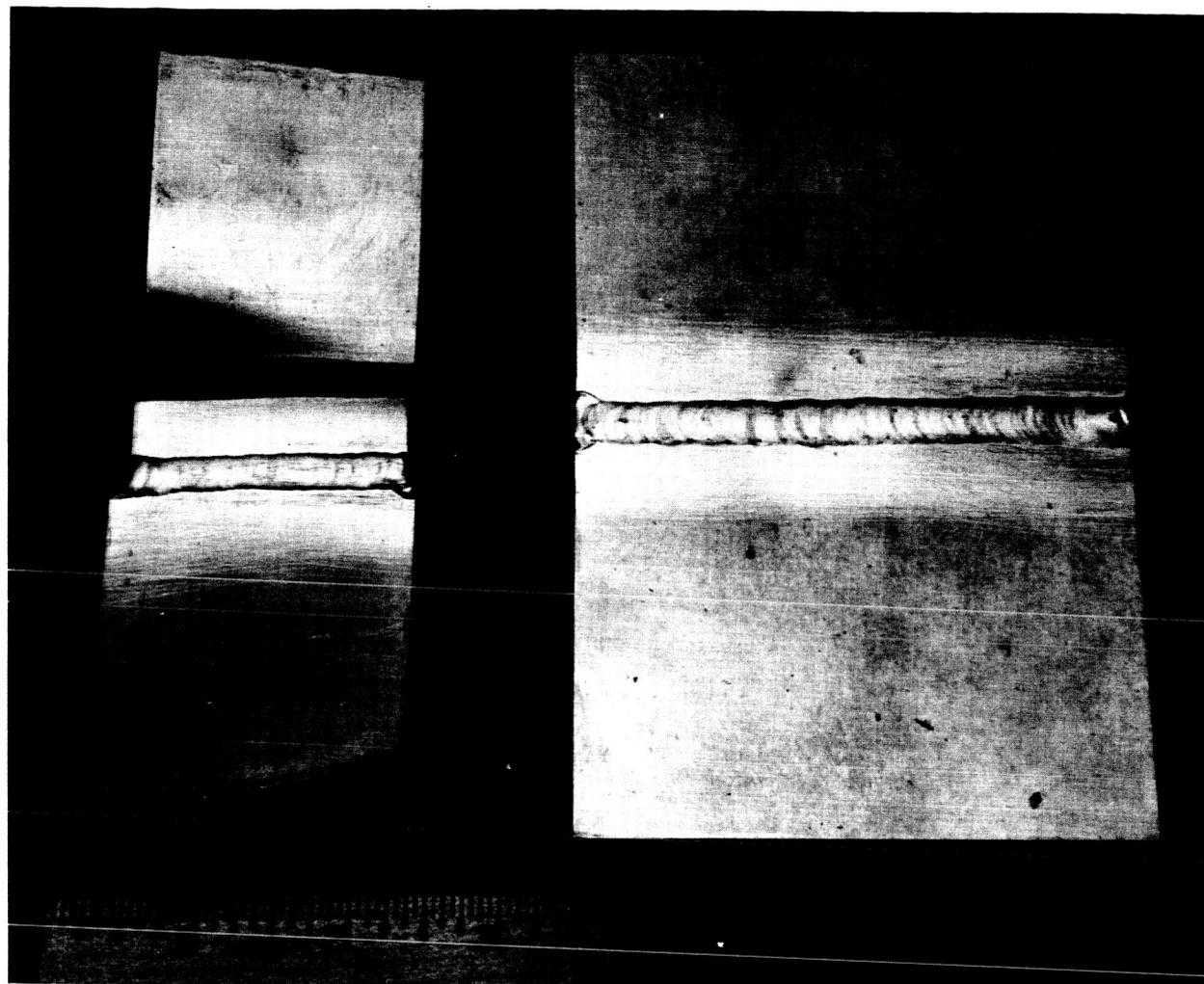
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Figure 54. Welded Panels Of Alloy 1A6 Showing A Test Panel That Broke Upon Impact In An Area Outside The Weld.

was not required. Electroplating the alloys was found to be an easy task.
This was done by applying an initial low temperature (120°F) electroless
nickel plate and flash plating it with copper prior to gold plating. The
composition of the electroless nickel bath is an follows:

$$50 \text{ g/l} \qquad NiSO_4 \cdot 6H_2O$$

$$25 \text{ g/l} \qquad NaH_2PO_2 \cdot H_2O$$

$$25 \text{ g/l} \qquad Na_4P_2O_7$$

The first two compounds were mixed with water at 150°F prior to dissolving
the sodium pyrophosphate. Water was added to make one liter and NH_4OH
added to the solution until a pH of 10 was obtained. No special surface prepara-
tion was found necessary prior to nickel plating which took a total of two minutes
to obtain a uniform coating. Copper flashing was found necessary for obtaining
a pit-free coating for the final metal coating. No corrosion was observed on the
gold coated specimens after four months of exposure to the atmosphere in a metal-
lography laboratory. The gold coating was applied in a cyanide bath and it was
found necessary to completely cover the specimen with electroless nickel before
copper flashing or a black deposit would result.

A very thin and hard plastic coating was successfully applied to a
specimen of alloy II 4 pilot ingot material. The surface of the coated panel
remained bright and shiny after one month's exposure in a metallography
laboratory and in a humidity cabinet.

The coating is called Sealtemt #630, a product of Rector Engineering
Company of Washington, D. C. , and is essentially a polyurethane coating which
cross links any surface moisture during air drying to stifle corrosion. The
coating contains xylol, cellosolve acetate (Union Carbide), and urethane; the
first two eliminate a blushing problem which might cause blistering at the
interface and promote good flow characteristics to keep the coating as thin as
possible.

The coating air drys in 4 hours at 70°F and 50% relative humidity
with a Swoard Hardness (NBS) of 25; after 3 days the hardness is 36 and
after 7 days it is 40. Another coating, an epoxy, called Castaband #250
which is very flexible in liquid nitrogen and inert to a hard vacuum, was
studied. There was no problem in applying this coating to the pilot ingot
material.

Specimens were successfully zincated by the MacDermid Corporation
and then copper flashed for subsequent plating. This company also tried to
anodize II 4 pilot ingot material but requested more material to develop a satis-
factory coating. They were successful in applying a chrome phosphate coating

which appears satisfactory but was received too late for testing.

Amchem Products Inc., of Michigan, have applied a chrome-phosphate conversion coating at 130°F called Alodine LFN-62-20. Specimens are being tested in Michigan and duplicate specimens have not as yet been sent to the AMF laboratory.

The NASA coating reported in NASA Tech Brief 65-10294, October 1965, which was modified for magnesium-lithium alloys, was successfully applied and conversion coatings are being developed by Allied Research Products of Maryland. The company has requested additional material for further work.

2.7 X-Ray Diffraction Studies

X-ray diffraction patterns were obtained for the modified II4 alloys early in the program and for the three alloys selected for scale-up melting and processing. Flat, polished specimens were placed in a rotating specimen holder which was part of the Norelco x-ray diffraction equipment used for these studies. The specimens were rotated at a speed of two degress per minute and irradiated with CuKα

The x-ray data obtained for the three wrought, pilot ingot materials are tabulated in Table 6, A through G, along with a pattern obtained for pure magnesium. The maximum intensity occurred at a 2θ angle of 36.10 to 36.15 degrees for alloy IA6 and did not change as a function of solution heat treating time or as a function of aging time. The maximum intensity occurred at the 101 plane, as it did for pure magnesium, with a corresponding d value of 2.485Å which also did not change significantly with heat treatment.

The maximum intensity also occurred at the 101 plane for alloy ZLH 972 with a corresponding d value of 2.491 Å. This alloy is actually a modification of alloy II4D which contained 8% zinc and 7% lithium. The pattern obtained for alloy II4D (not shown) was similar to the one obtained for the unmodified alloy II4 shown in Table 6E. For this alloy the maximum intensity occurred at a 2θ value of 35.1 degrees after rolling and prior to heat treatment with a corresponding d value of 2.556. Upon solution heat treating and aging, the pattern changed significantly. The maximum intensity occurred at a 2θ value of 51.35 degrees with a corresponding d value of only 1.779Å. This plane has tentatively been identified as 003 indicating that the mechanism of aging is vastly different in alloy II4 compared with the other two major alloys developed in this program. A great deal more work needs to be done to obtain quantitative results from the x-ray studies. The data in Table 6 are presented as a reference until these studies are complete. A series of alloys with varying percentages of lithium will have to be made in order to obtain magnesium standards in the α + β range. The aging characteristics of alloy II4 must

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be studied extensively especially in the initial stages of aging during the formation of Brillouin-Preston Zones.

A total of 9,933 man-hours was expended from June 19, 1965 through March 20, 1966.

TABLE 6

X-RAY DATA

A. Pure Magnesium 99.9999

2θ	d value	Intensity %	hkl
69.9	1.36	8.65	201
68.5	1.369	27.8	112
63.1	1.473	11.15	103
47.8	1.903	22.25	102
36.6	2.452	100	101
34.4	2.607	79	002
32.1	2.788	49.4	100

B. Alloy I A 6, Rolled at 260°F, Solution Heat Treated 1½ Hours at 600°F and Aged 20 Hours at 200°F

2θ	d value	Intensity %	hkl
87.8	1.112	11.7	
80.75	1.190	10.9	
76.55	1.244	17.5	
73.2	1.293	12.1	
68.3	1.373	32.0	112
64.8	1.439	85.7	
64.0	1.455	15.4	
57.75	1.596	13.6	110
54.6	1.681	22.5	
52.0	1.758	53.0	
48.4	1.881	14.3	
44.75	2.025	37.0	
37.8	2.380	39.5	
36.85	2.442	26.6	
36.15	2.485	100	101
35.15	2.553	22.8	
32.35	2.767	27.6	100
32.95	3.875	67.8	

C. Alloy I A 6, Rolled at 260°F, Solution Heat Treated 3 Hours at 600°F and Aged 1 Hour at 200°F

2θ	d value	Intensity %	hkl
76.6	1.244	10.4	
73.05	1.295	13.0	
69.5	1.352	10.4	
68.3	1.373	20.0	
64.9	1.436	70.0	
64.15	1.452	17.4	
57.8	1.570	14.6	
54.6	1.681	22.8	
52.0	1.758	72.0	
48.5	1.877	14.1	
44.7	2.027	24.6	
43.3	2.089	14.4	
40.2	2.243	18.4	
37.85	2.380	30.0	
37.0	2.429	26.2	
36.15	2.485	100	101
35.1	2.556	32.0	002
32.4	2.763	21.6	100
26.5	3.333	28.0	
24.35	3.655	32.4	
23.0	3.867	74.2	

D. Alloy I A 6, Rolled at 260°F, Solution Heat Treated 3 Hours at 600°F and Aged 20 Hours at 200°F

2θ	d value	Intensity %	hkl
87.7	1.120	14.7	
76.5	1.245	14.9	
73.0	1.296	15.3	
68.3	1.373	23.3	112
64.8	1.439	51.5	
64.2	1.451	21.7	
57.7	1.598	18.2	
54.55	1.682	33.7	
51.9	1.762	87.0	
48.4	1.881	20.2	102
44.65	2.029	29.4	
40.15	2.246	23.7	
37.8	2.380	37.7	
36.95	2.443	30.0	
36.10	2.488	100	101
35.1	2.556	38.8	002
32.3	2.771	38.8	100
26.4	3.376	36.4	
24.2	3.677	45.5	
22.85	3.892	89	

E. Alloy II 4 As Rolled at 450°F

2θ	d value	Intensity %	hkl
87.7	1.113	7.2	
83.05	1.163	14	
74	1.281	11	
68.95	1.362	15.5	112
64.95	1.436	46	
65.15	1.452	37.5	
57.8	1.595	11.5	110
55	1.669	17	
52.1	1.755	47	
48.5	1.893	21.5	102
45.05	2.012	22.5	
42	2.151	13.5	
40.1	2.248	16.2	
38.1	2.365	15.5	
37	2.429	54	
36.15	2.485	29	101
35.1	2.556	100	
32.45	2.759	18.5	
30.75	2.907	21.7	
26.75	3.333	22.7	
23.2	3.834	80.7	
20.75	4.281	29.8	

F. Alloy II 4, Rolled at 750°F, Solution Heat Treated 3 Hours at 600°F and Aged 20 Hours at 200°F

2θ	d value	Intensity %	hkl
87.1	1.118	32.7	
68.8	1.363	32.7	201
64.2	1.451	42	
63.4	1.467	30.7	103
57.1	1.613	58.5	
54.3	1.689	37.5	
51.35	1.779	100	003
39.4	2.287	27	
36.3	2.475	48.5	101
35.45	2.537	63	
34.4	2.607	46.8	002
22.35	3.978	42.2	

G. Alloy ZLH 972, Rolled at 450°F, Solution Heat Treated 1 Hour at 800°F and Aged 1 Hour at 200°F

2θ	d value	Intensity %	hkl
87.6	1.114	8.75	
76.6	1.244	7.5	
73.7	1.285	8.45	
70.55	1.335	8.75	
69.25	1.357	11.75	
68.75	1.365	13.7	112
64.8	1.439	20	
64.0	1.455	16.9	
60.3	1.535	8.0	
57.75	1.596	20	110
54.9	1.672	47.2	
51.9	1.762	100	
48.4	1.881	20.2	102
44.95	2.017	25.7	
38.05	2.365	51.8	
36.85	2.439	67.5	
36.05	2.491	93.8	101
34.9	2.571	34.3	
32.3	2.771	25.3	100
26.65	3.346	30.0	
23.05	3.858	40.6	

3.0 REFERENCES

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3. W. R. D. Jones and G. V. Hogg, "The Stability of Mechanical Properties of Beta-Phase Magnesium-Lithium Alloys", J. Inst. of Metals 85, 1956-57, 255.