NAVAL POSTGRADUATE SCHOOL Monterey, California

THESIS

HIGH STRENGTH ALUMINUM-MAGNESIUM ALLOYS THERMOMECHANICAL PROCESSING MICROSTRUCTURE AND TENSILE MECHANICAL PROPERTIES

by

Raymond Arthur Grandon

March 1979

Thesis Advisor:

T. R. McNelley
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Thesis G6553

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High Strength Aluminum-Magnesium Alloys: Thermomechanical Processing, Microstructures and Tensile Mechanical Properties

by

Raymond Arthur Grandon, Jr. Lieutenant Commander , United States Navy B.S., University of Nebraska, 1966

Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN APPLIED SCIENCE

from the

NAVAL POSTGRADUATE SCHOOL

MARCH 1979

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ABSTRACT

Microstructures and mechanical properties of thermomechanically processed Aluminum- Magnesium alloys were investigated in this research. Magnesium content of the alloys ranged from ⁷ to 12 weight percent and an alloy containing 10 percent Mg and 0.5 percent Cu was also examined. Thermomechanical processing treatments involved solution treatment followed by warm isothermal rolling. Temperature for this warm rolling was typically 300° C, and this is below the solvus temperature for the alloy. Such processing results in a fine dispersion of the intermetallic compound Al_3Mg_2 (8) in a solid solution matrix. Typical mechanical properties are an ultimate tensile strength of 520 Mpa (75000 psi) , with 12 percent elongation to fracture. Such a material may be further cold worked to ultimate tensile strengths of 620 Mpa (90000 psi) with ⁶ percent elongation to fracture. Dynamic recrystallization in such alloys was also studied; control of recrystallization is necessary to achieve a uniform dispersion of the intermetallic in subsequent processing.

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I. INTRODUCTION

It is difficult to imagine our modern world without aluminum. From space craft to beverage cans, aluminum has been chosen as the material for fabrication. So many uses have been found that aluminum ranks second only to steel in tonnage produced yearly. What are the properties of this metal which enable it to find such wide usage? Every application in which aluminum is used makes use of one or more of the following properties

1. Density.

At room temperature, pure aluminum has a density of 2.7 grams per cubic centimeter; excepting magnesium, it is the lightest of structural metals. For the same weight, it will cover about three times the surface area as steel or copper of the same thickness [Ref. 1].

2. Strength.

The strength and hardness of aluminum alloys do not approach those of higher strength and hardness steel; however, many aluminum alloys equal or exceed the strength of plain carbon steels and must be considered as alternative materials. Heat treated aluminum alloys are available in tensile strengths above 80 KSI.

3. Ductility.

The face-centered cubic crystal structure of aluminum allows it to be easily drawn, forged, stamped, rolled or

otherwise worked at low temperatures, with low-power equipment and low wear on the equipment. In contrast, steels often must be worked at high temperatures with high-power equipment and considerable wear on the equipment.

4. Corrosion Resistance.

The ability of aluminum to form a highly corrosionresistant oxide is one of its frequently exploited features. Annodizing is a process which increases the thickness of the oxide film and further increases aluminum's natural resistance.

Aluminum alloys may be classified as either wrought or cast materials. Wrought aluminum alloys presently in use can be further classified as either heat treatable or nonheat treatable. The compositions and tensile properties of several common wrought aluminum alloys are given in Table I. The alloys which are the subject of this thesis would be classed as wrought. The alloys which do not require heat treatment subsequent to forming are less expensive to produce and to use.

Many of the aluminum alloys presently in production have magnesium as their primary alloy addition. If magnesium is the principal alloying element in a wrought alloy, it will have a four-digit, 5000 series designation, e.g., 5052. Cast alloys have a three-digit designation system. Magnesium is used as an alloying agent for several reasons [Ref. 1]. It is an effective hardening agent in solid solution. For example, 0.8 weight percent Mg is equivalent to 1.25 weight percent manganese. It is more soluble in solid solution than manganese,

Table I

Mechanical Properties of Several Principal Aluminum Alloys as 1/16 In-Thick Sheet (Adapted from Reference 2)

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or any other solid solution strengthener . Magnesiumcontaining alloys have excellent welding characteristics; in addition, such alloys are corrosion resistant in marine environments and also shock and fatigue resistant. Magnesium is the only widely used alloying element which is less dense than pure aluminum, and its addition to aluminum results in an alloy less dense than pure alunimum. The densities of some common commercial alloys are listed in Table II.

TABLE II

DENSITIES OF SELECTED ALUMINUM ALLOYS (Adapted from Reference 3)

II. THERMOMECHANICAL PROCESSING

A. BACKGROUND

Thermomechanical processing of a metallic material is generally intended to shape the material and refine its microstructure to enhance mechanical and physical properties for its intended use. Cold rolling and hot rolling are common thermomechanical processes and knowledge of their effects on materials is well advanced. The kind of thermomechanical processing used in this thesis does not fit well within the bounds of either classification but rather falls between, and hence is described aswarm rolling. The effects of warm rolling on microstructure are less well known, primarily because it is not, at this time, a commercial process. Interest in this area was stimulated by Sherby [Ref. 4] when he reported the development of a warm rolling process for high-carbon steel, which resulted in a material of good ductility and high strength, something not generally thought possible under the rules of hot or cold rolling.

The highest strengths in commercial aluminum alloys are currently accomplished using heat treatable alloys like 7075. This alloy uses the combined effects of solid solution and precipitation hardening to achieve its strength. Non heattreatable aluminum alloys utilize solid solution hardening and possibly cold working to achieve their strength, but are considerably weaker than the strongest heat-treatable alloys.

Dispersion hardening is similar to precipitation hardening in that it utilizes the strengthening effects of a finely dispersed second phase [Ref. 5]. A difference lies in the coherency of the second phase; in dispersion hardened materials, the second phase is incoherent with the matrix, while in precipitation hardened materials the second phase is coherent. Dispersion hardened materials are produced by mixing an insoluble second phase in a fine, particulate form with a metal, either in liquid or powder form. The degree of hardening realized from a second phase depends on its distribution, volume fraction, and particle size and shape. This method is exacting, expensive, and therefore, little used.

A strong aluminum alloy could be produced if one could combine the effects of strain and dispersion hardening. This thesis is an attempt to demonstrate this and warm working, by rolling, is the method used to achieve a high-strength wrought aluminum-magnesium alloy. Previous work in this area has been done by Ness [Ref. 6], Bingay [Ref. 7], and Glover [Ref. 8] at the Naval Postgraduate School.

B. PREVIOUS WORK

Ness [Ref. 6] studied an 18% magnesium alloy and demonstrated that a warm rolling process could produce an alloy with a homogeneous microstructure and compressive strength in excess of 95 KSI. The alloy used by Ness [Ref. 3] was made by him at the Naval Postgraduate School, and the quantities of material produced were small and insufficient to allow for tensile testing. The rolling procedure employed by Ness entailed very

 $\Delta \sim 10^{11}$
slow rolling, with small reductions per pass and long reheating times between passes.

Bingay [Ref. 7] and Glover [Ref. 8] followed the work of Ness [Ref. 6]. Because of difficulties in compounding relatively large quantities of these alloys in the laboratory, a commercial source was sought and found in Kaiser Aluminum and Chemical Corporation. Bingay [Ref. 7] attempted to achieve microstructural refinement of alloys containing 15 and 19 weight percent magnesium, through warm upset forging, both isothermally and non-isothermally. This processing entailed much higher deformation rates than those used by Ness. Neither method achieved the microstructure produced by Ness, and Bingay concluded that the as-cast alloy was not suitable at these magnesium contents for processing under such more practical conditions. It was recommended that alloys below 15% Mg be used and that solution treatment before mechanical processing be conducted. This would allow for concurrent working during precipitation from solid solution. Glover [Ref. 8] worked with the alloys produced by the same source as Bingay's, and directed his efforts toward using forged material to produce rolling material, upon which tensile testing could be done. Again, no problems were encountered in forging the 15 and 19% material, but attempts at subsequent rolling (at relatively high rates) failed due to cracking. Because of this Glover shifted his efforts to an alloy thought to contain 11% Mg. This material was found to be rollable after forging and material for tensile testing was obtained. Ultimate tensile strengths slightly above 60 KSI, were attained, with

elongations of 4-6%. Glover reported his results as related to a 7% Mg alloy because various quantitative analyses of the 11% material indicated this to be closer to the actual magnesium content.

C. PURPOSE OF THESIS

The work of Bingay [Ref. 7] and Glover [Ref. 8] raised a great many more questions than it answered and served to demonstrate how little was known about the effects of warm thermomechanical processing on aluminum-magnesium alloys of such high magnesium content.

This research has taken up the suggestions of Bingay [Ref. 7] and Glover [Ref. 8], namely that alloys with magnesium content below the maximum solubility of magnesium in aluminum be used, and that these alloys be initially heat treated to a fully solution-treated condition prior to processing. This study will focus upon the effects of warm rolling on such alloys and demonstrate that such processing can be utilized to produce a high-strength alloy.

III. PROCESSING PROCEDURES AND EXPERIMENTAL METHODS

A. PROCESSING EQUIPMENT

Materials were solution treated and heated for rolling in a Blue M Model 8655F-3 Stable Glow Box Type furnace with a proportioning control system. For isothermal rolling billets were reheated, using this furnace, between each pass through the rolling mill.

All rolling was done on a 2-high, variable-speed rolling mill, Model 172, manufactured by Fenn, Inc. The maximum capacity of this mill was material 1.25 inches (32mm) in thickness and ⁶ inches (152mm) wide. Roll diameter is 4.25 inches (108mm)

B. MECHANICAL TESTING EQUIPMENT

Hardness testing was done on a Wilson Model 1JR Rockwell Hardness Tester, using the Rockwell "B" scale. Tensile testing was done on a Model TT-D Instron Floor Model Testing Machine. A constant crossheat speed of 0.05 inches (1.27mm) per minute was used for all testing.

Specimen blanks for tensile testing were $4.7 \times .75$ inches (120 x 19mm), cut from rolled plate. Gage length was 1.5 inches (38mm), unless otherwise noted. Elongations were based upon the measured extension at fracture. The engineering stress-strain curves are based upon data obtained from the Instron tensile testing machine. The stress-strain curves were adjusted to reflect actual measured elongation at fracture.

C. METALLOGRAPHY

Standard methods were used to produce specimens suitable for microscopy. Two different etching procedures were used to bring out the microstructural features of interest. One utilized a dilute solution of warm phosphoric acid and was excellent for examining beta phase distribution. The other utilized dilute fluoroboric acid (Barkers Reagent) and electrolytic etching of the specimen. This produced excellent results for determining grain size. These procedures are described in Ref. ⁹ under the designation of etchants eight and five, respectively. Micrographs using method two are made with crossed polarizers in an optical microscope.

Light micrographs were made on a Bausch and Lomb Balplan microscope. Polarized incident light was used for micrographs of material etched with phosphoric acid. Crossed polarizer and analyzer were used for micrographs etched fluoroboric acid (Barkers Reagent) . Scanning Electron Micrograph were made with a Cambridge Scientific Instruments Model S4-10 Stereo Scanning Electron Microscope.

IV. PROCESSING VARIABLES

A. SOLUTION TREATMENT

The alloys used in this study were produced by the direct chill casting method at the Alcoa Technical Center, Alcoa Center, Pennsylvania. In direct chill casting molten metal is poured into the top of a water-cooled copper mold and solid material is withdrawn from the bottom. The bottom end of the mold is initially closed by a water-cooled copper plate, and this plate is then lowered as a support for the solidifying alloy. This method provides a high cooling rate which is needed to curtail segregation in the alloy. The microstructure which results is uniform on a macroscopic level, but on a microscopic scale it is dendritic and thus non-uniform.

Solution treatment, i.e. heating for a period of time at a temperature at which a single-phase solid solution is stable, is the process by which microscopic differences in composition are reduced or eliminated. Solution treating requires heating to a temperature above the solvus for the alloy and thus in the single phase region denoted α on the partial aluminum-magnesium phase diagram, Figure 1.

Bingay [Ref. 7] and Glover [Ref. 8] did not utilize solution treating in their earlier work, it being thought that mechanical working at warm temperatures would lead to homogenization by recrystallization and strain-enhanced

diffusion. Subsequent work by them demonstrated at best only a partial homogenization and thus relatively poor mechanical properties and difficulty in processing, especially in the 15% and 19% magnesium alloys.

Figure 2(a) shows the as-cast, dendritic microstructure of a 12% Mg alloy. The beta phase is coarse and follows a dendritic pattern. A study was conducted on this 12% Mg (57 series) alloy to determine how much time at the solution treating temperature was required to dissolve the beta phase. Cubes, 0.5 inch (13 mm) on a side, of this alloy were solution treated at 440°C for times of 5, 10, 20, 40 and 60 hours. Selected micrographs of this study are shown in Figure 2. The beta appears to have completely re-dissolved after ⁵ hours and at times above 20 hours little further charge can be detected; hence, 20 hours at 440°C was selected as a standard solution treatment to be used in this study.

The temperature of solution treatment used in the preceding experiment was selected for two reasons: first, burning must not occur and second, nearly complete resolutioning at reasonable times must occur. Since the beta phase melts at 451°C, this temperature must not be exceeded. Second, 12% magnesium is soluble in aluminum only above 410°C, and this temperature must be exceeded for complete solutioning. 440°C was selected to provide room for error in temperature control and to speed the process as much as possible.

 (c)

 (d)

Micrograph (a) shows the dendritic non-homogeneous
as-cast structure of a 12% Mg alloy. Micrographs
(b), (c) and (d) show the effects of solution
treatment at 440° C for 5, 20 and 60 hours, Fig. $2.$ respectively. 100X, prepared by method 1.

To preserve the homogeneous microstructure obtained by solution treatment, a high cooling rate such as obtained from water quenching is frequently employed. Water quenching of rolling billets containing more than eight percent magnesium was found to cause cracking. A study of microstructure resulting from air cooling revealed that the rate of cooling by this method was sufficient to prevent re -precipitation of the beta phase; therefore, air cooling was adopted for alloys containing more than ⁸ percent magnesium.

B. ROLLING TEMPERATURE

Many and sometimes conflicting factors must be considered in rolling temperature selection. To obtain a fine-sized second phase particle, lower temperatures are necessary to minimize growth. The total amount of second phase is increased at lower temperatures, however, given a particular magnesium content. Figure ³ is a plot of the equilibrium volume fraction of beta, based on the Lever Rule, as a function of temperature for several magnesium contents. How much beta phase is in the final microstructure is not directly obtainable from this figure because processing is not done under equilibrium conditions. The curves of Figure 3 do indicate the maximum volume percentage of beta obtainable at a temperature. Normally, the ductility of a metal will increase and its strength will decrease as temperature increases, making rolling easier. This favors a high rolling temperature. However, as the melting range of an alloy is approached,

ig. 3. These curves reflect the volume percentage of an Al-Mg alloy microstructure which would be Beta phase, or the intermetallic compound Al₃ Mg₂, if the system were at equilibrium.

hot shortness, cracking due to segregation of alloy elements to grain boundaries, begins to occur, setting an upper limit on rolling temperature. Strength and ductility are strongly dependent upon grain size; particular attention must therefore be paid to the way it is affected by the rolling temperature. Pure aluminum has a high stacking fault energy as indicated by the ease with which cross slip and climb occur. This prevents misorientation of subgrains, formed during deformation, from reaching the extent required for recrystallization. Rolled pure aluminum often has an elongated grain structure; this also occurs in warm-rolled aluminum-magnesium alloys. Magensium addition to aluminum lowers the stacking fault energy and may allow for recrystallization to occur during rolling at an intermediate temperature. Determination of the required temperature is the subject of an experiment to be discussed later. Grain growth is an undesirable phenomena favored by higher temperatures; therefore, a balance must be reached between recrystallization and grain growth. Figure 1 depicts rolling temperature as a function of the aluminum magnesium phase diagram. Tables III and IV contain a complete listing of the rolling temperatures used.

C. ROLLING SCHEDULE

The term rolling schedule, used throughout this thesis, refers to the rolling reduction pattern employed to process

the alloy in question. Rolling consists of passing a piece of material between parallel, rotating steel cylinders of separation less than the thickness of the piece. The workpiece is plastically deformed and reduced in thickness by this process. The effect of this on the microstructure and properties of the material being processed is dependent on the temperature of the workpiece, the total strain and the strain rate during rolling. When rolling is done at warm and hot temperatures, strain rate during rolling becomes important. Ford and Alexander [Ref. 10] derived the following equation for the mean strain rate ϵ experienced by the material during a rolling pass:

$$
\frac{1}{\varepsilon} = \frac{v_r}{\sqrt{Rh_0}} \sqrt{r} (1 + \frac{r}{4})
$$
 (1)

where v_r = 2 π Rn is the speed of the roller surface, R is roller diameter and n is the rotational speed of the rollers in revolutions per second; h_{0} is the initial thickness of the workpiece, h_f is the final thickness, and $r = (h_o - h_f)/h_o$ is the fractional reduction during the rolling pass.

The factors listed above as variables may be reduced. For the purposes of this study, only one size of roller, 4.25 inches (10.41 cm), and only one rolling speed, 0.267 revolution per second, were used. The variable r is easily controlled within the power limitation of the rolling mill. The rolling schedules used during most of this thesis

were selected on the basis of either fixed percentage reduction or fixed $h_{\alpha} - h_{f}$ per pass. Calculation of the resulting strain rates, based on equation (1), was done and is shown graphically in Figure 4, as a function of pass number. The data here is for the rolling pattern employed with an alloy of 7% Mg, to be subsequently discussed.

D. NON-ISOTHERMAL PROCESSING CONDITIONS

It will be noted in many of the figures and tables of this thesis that rolling conditions are not all isothermal. There were attempts to duplicate the patterns used by Sherby [Ref. 4] in the processing of high-carbon steel. His process calls for rolling through a phase transformation to achieve a fine, homogeneous microstructure. It was thought that a similar effect might be produced by rolling through a temperature range for the aluminum-magnesium alloy wherein recrystallization would occur.

The non-isothermal condition used in these experiments consisted of making multiple rolling passes without reheating between passes. This resulted in each successive pass being made at a lower temperature. If recrystallization should occur during this process, the homogenizing effects of recrystallization with concurrent straining would be realized. The equipment imposed a limit on the rate at which passes could be made. The best rate achieved was 12 per minute. Instrumentation to monitor temperature during the actual rolling processes was not available; however, an experiment was devised to provide a guideline on temperature behavior

lig. 4. This figure depicts the average strain rate resulting from rolling schedules used to process 7% Mg alloys. Rolling billets had an initial thickness of .75 inches. Curves were calculated assuming hot rolling conditions; the letters A, B and C refer to a 7% Mg alloy rolled at 300 $^{\circ}$ C, and D, E, and F, at 250 $^{\circ}$ C.

during rolling. A small hole was drilled into the center, from the end, of a standard rolling billet. A small thermocouple was fitted into this hold. Subsequently, the billet was heated to 460° C and rolled at about 5 passes per minute, to a thickness of .5 inches (1.27 cm) . The temperature was recorded during this process. Analysis of this data indicated a rate of temperature drop of 100°C per minute. Rolling without the thermocouple can be accomplished with a larger number of passes per minute; nonetheless, the time out of the furnace and in contact with the run-in and run-out tables and the rollers, appeared to be the main factor and thus the same rate of temperature decrease is assumed to apply.

V. SEVEN PERCENT STUDY

The alloys ordered from Alcoa were not available for study during the first half of the research period. In preparation for work to be conducted upon them, a series of experiments were conducted on the seven-percent magnesium alloy used by Glover. The intent of this study was to determine the effects of solution treatment, rolling temperature and rolling schedule.

A. PROCEDURE

Six rolling billets, $.75 \times .75 \times 2.0$ inches (18 \times 18 \times 50 mm), were solution treated for 20 hours at 440° C and water quenched. From Figure ¹ it can be seen that this temperature is well within the solid solution range for a 7% Mg alloy. Three rolling schedules were devised to determine strain rate effects and the extent of ductility of the material. These schedules were 5% per pass, 10% per pass and 10% for ⁵ passes then 25% per pass to final thickness. Total strain was to be 2.5. The strain rate as a function of the number of the pass for each of these schedules is shown in Figure 4. As noted previously, for larger reductions per pass fewer passes are required and the material will experience less time at temperature. Rolling temperatures were 250°C and 300°C, 65 and 15 degrees, respectively, below the solvus temperature for this alloy.

$B -$ RESULTS

Solution treatment produced a major change from the results previously obtained with this alloy. All billets were rolled successfully, and with no evidence of cracking. Tensile specimens were prepared from each of the rolled billets and tensile tests conducted; the results are shown graphically in Figure ⁵ and presented in Table III. Selected micrographs of the asrolled materials are shown in Figure 6.

C. DISCUSSION

The data of Figure ⁵ suggest that low rolling temperature and high strain rate during rolling lead to increased strength and reduced ductility. Notable is the large ductility associated with condition A, the material rolled at the slowest rate and highest temperature. In this case, microstructural analysis, described below, indicates partial recrystallization occurred during processing.

Generally, recrystallization did not occur during the thermomechanical processing, resulting in a banded microstructure with precipitation primarily along elongated grain boundaries. Specimen A, rolled at 300° C, shows partial recrystallization and experienced a longer time at processing temperature than with B or C (also rolled at 300° C). All three samples were allowed 30 minutes to warm up; due to the different rolling schedules, specimen A was at temperature approximately 190 minutes; B, 100 minutes; and C, 60 minutes. These different total times at temperature span a range, the upper end of which is sufficient for recrystallization to begin. Specimen A, being only partially recrystallized, is an

TABLE III

THERMOMECHANICAL PROCESSES AND MECHANICAL TESTING RESULTS (7% Mg ALLOY)

TABLE KEY

Solution treated; XXX/XX: Temperature (degrees Centigrade)/time (hours)
Air cooled ST:
AC:
WW:

Warm worked; XX/XXXX: Percent reduction per pass/number of passes/
temperature (degrees Centrigrade)

ig. 5. Engineering stress-strain curves for the 7% Magnesium Alloy. Comparison of these samples with the same reduction schedule but different temperatures isolates the effect of process temperature, while comparison of these samples within the same temperature group isolates the effect of reduction schedules.

(c) (d)

Fig. 6. Micrographs (a) and (b) depict longitudinal sections of specimen A; (c) and (d) depict longitudinal sections of specimen F. The partial recrystallization of A can be seen in micrograph (b). (a) and (c) $400X$ prepared by method $\overline{1}$, (b) and (d) 100X, prepared by method 2.

indication that at a 300 $^{\circ}$ C rolling temperature, recrystallization is possible but not a rapid process. It is slow to the extent that the time allowed for specimens B and C was not sufficient. Recrystallization at the lower temperature of 250° C is so slow as to not have occurred even for the longest time of exposure.

Slight grain growth during solution treatment did not prove to be detrimental to the rolling process. At 300 $^{\circ}$ C, growth undoubtedly occurred, but the very large grain size of the as-cast material made its effect proportionately small. The solutioning of the beta phase produced a major improvement in rollability of the material and far offset any detrimental effects of grain growth. The rate of grain growth during rolling of the material was not a factor which could be determined because of the general lack of recrystallization and the elongated grain structure. Although the solution treatment used appears to be sufficient in this experiment, this does not mean that sub-microscopic variations in composition were eliminated, only that their effects were reduced to a point where they posed no difficulty.

The strengthening effect of higher reduction schedules must have been the result of a more highly refined grain substructure. The lower reduction rolling schedules imply lessened rates of dislocation generation and allow diffusion more time for their elimination. This effect would account for the strength differences of specimens with the same rolling temperature and where recrystallization did not occur.

Between temperature groups this would also be a factor, indicating higher strengths at lower processing temperatures because diffusion rates are decreased as temperature decreases.

The amount and distribution of beta phase in the specimens did not appear to vary in a quantifiable manner in specimens B-F. The size of second phase precipitate particles vary widely within each of these samples but in about the same amount in each, indicating temperature did not play a major part in their size and distribution. Specimen A appears to have the same variety of particle sizes but the distribution is significantly more homogeneous, a probable result of recrystallization. Rolling schedule does not appear to be a factor in determining particle size and distribution.

VI. TEN PERCENT STUDY

A. BACKGROUND

The alloys studied in this series of experiments were obtained from Alcoa, as previously discussed. Primary emphasis was on alloys containing 10.2% magnesium. Those specimen designations beginning with the number 51 contain no alloying addition other than Mg. Those designations beginning with the number 54 contain 0.5% copper in addition to 10.2% magnesium. The selection of alloys with this magnesium content was based upon the recommendations of Bingay [Ref. 7] and Glover [Ref. 8] and the desire to work from a basis established by the study on the 7% Mg alloy.

The overall objective remained the production of a highstrength aluminum magnesium alloy, but as was discovered early in the study, strength in excess of 80 KSI was obtainable in a straight-forward thermomechanical process; the objective then became the refinement and improvement of the microstructure.

$B.$ MATERIALS

The rolling billets used in these experiments had the dimensions 1.2 inches (3.05 cm) square by 3.0 inches (7.62 cm) in length, cut from chill-cast materials. Figure ⁷ illustrates the microstructure of the material in an as-cast and as solution-treated condition. The microstructure is dendritic and coarse grained; solution treatment removes

 (a) (b)

 (c) (d)

 $_{\rm Fig.}$ 7. Micrographs (a), (b) and (c) show the structure of alloys 51, 54 and 57, respectively, after solution treatment at 440°C for 20 hours. (d) shows the as-cast structure of a 57 (12% Mg) series alloy. 100X, prepared by method 2.

the non-equilibrium beta phase but leaves a coarse, irregular grain structure.

C. BASIC PROCESS

Isothermal rolling at 300°C following a 20-hour solution treatment at 440°C, was established for the 51 and 54 series of alloys as a baseline in determining the effects of process variation. Isothermal rolling was accomplished by an initial series of passes at a 5% reduction per pass followed by finishing passes at 10% reduction per pass; the mean strain rate during processing is illustrated in Figure 8. The material was reheated between each pass and rolled to a true strain of approximately two. Specimens 512 and 541 are the result of this process. The tensile data and micrographs from these two materials, as processed, were then used for comparison of results from variations on this basic process. Figures ⁹ through 12 show micrographs of the structures and the engineering stress-strain curves of specimens 512 and 541.

D. DIFFERENCES IN BASES

The two basis materials themselves are strikingly different and the differences are directly attributable to the 0.5% copper addition to the 54 series alloy. Grain size is difficult to determine in the 541 specimen because of the large apparent amount of uniformly distributed second phase. Matrix grains in the 512 alloy (no copper addition) are highly elongated in the rolling direction and the beta

Fig. 8.

Average strain rate as a function of the number of passes during rolling for billets initially 1.2 inches thick. These curves are based upon equations used to calculate average. strain rate in material being hot rolled and are for the rolling schedules used to process the 10.2% Mg alloys

Fig. 9. A dramatic increase in tensile strength is caused by the addition of .5% copper. The thermomechanical processing of these samples was the same.

 (a) (b)

(c)

Fig. 10. Micrographs (a) and (b) are cross-sectional views of specimen 512; (c) and (d) are longitudinal views. (a) and (c) are 400X prepared by method 1; (b) and (d) are 100X prepared by method 2.

Fig. 11. These micrographs illustrate the structure of specimen 541. (a) and (b) are cross-sectional views; (c) and (d) longitudinal views. (a) and (c) 400X, prepared by method 1, (b) and (d) 100X, prepared by method 2.

 (a) (b)

Fig. 12. (a) and (b) are SEM micrographs of a longitudinal section of specimen 512. (c) and (d) are SEM micrographs of specimen 541 in the same view, (a) 2500X, (b) 6200X, (c) 2700X, (d) 6600X, prepared by method 1.

 (c) (d)

phase is primarily in the matrix grain boundaries. It is believed, based on more detailed examination of the 541 alloy (with the copper addition) that the matrix grain structure in it is also elongated, perhaps to a lesser extent than in the 512 material. In addition to the beta phase intermetallic compound, $\texttt{Al}_3 \texttt{Mg}_2$, the micrographs show what appears to be a third phase, as an evenly distributed, larger precipitate. It appears in both specimens that recrystallization did not occur.

E. EFFECTS OF COLD WORK

Because recrystallization did not occur in the materials subject to this basic process and because it is considered necessary to produce a homogeneous, fine grained microstructure, specimen 511 was cold rolled during its 5% reduction step, a process sometimes used in other rolling systems to induce recrystallization. After the initial cold rolling, it was then heated and warm rolled to completion normally. All other processing variables were maintained as before in specimen 512. The results of tensile testing are shown in Figure 13 and in Table IV. Strength increased substantially while ductility decreased. The resulting microstructure is shown in Figure 14. Comparison with the micrographs of 512 show little change has occurred. It was noted that the amount of precipitate seemed to be greater near the rolled surface of 511, where greater straining occurred during cold rolling, than was observed in other samples subject to cold working prior to warm working. The objective of the experiment was not realized, however, in that recrystallization is not apparent.

Fig. 13. Engineering Stress Strain curves for 10.2% Magnesium Alloys. The 511CR and 512CR samples were made from samples 511 and 512, respectively. Comparison of 511 with 511CR and 512 with 512CR demonstrates the effect of cold rolling as a final process. Comparison of 511 with 512 shows the increase in strength resulting from cold rolling prior to warm rolling. Sample 514 is shown to demonstrate the effect of 20 hours of additional solution treatment at 490°C.

 (c) (d)

Fig. 14. The effects of cold rolling on 51 series alloy before and after warm rolling; (a) and (b) show the cross- sectional and longitudinal views of specimen 511; (c) and (d) show the same material after cold working. 100X, prepared by method 2.

TABLE IV

Table of Thermomechanical Processes Mechanical Testing Results (10.2% Mg Alloys)

Table Key

 $\bar{\nu}$

Small rolling billets were made of material from specimens 511 and 512, and designated 511CR and 512CR , respectively, These billets were given further cold work to a true strain of 2.8 at a reduction rate of 10 percent per pass. Tensile tests of these materials demonstrated that cold rolling could be done on this material and that it produced an increase in UTS in excess of 20 KSI. Figure 13 illustrates the engineering stress-strain curves obtained and Figure 14 illustrates the microstructure. No dramatic change is seen between the microstructures of 511 and 511CR or 512 and 512CR. Precipitation seems to have increased and its distribution appears to be more homogeneous. Of particular note is the result for material designated 511CR. The yield strength and ultimate tensile strengths obtained, 79 KSI (544 Npa) and 88 KSI (606 Npa) compare well with the tensile properties of high strength precipitation hardening alloys such as 7075. Notable as well is ductility, 6% elongation to fracture, also comparable to 7075.

F. EFFECTS OF ADDITIONAL SOLUTION TREATMENT

The distinctively banded nature of the microstructure in 511 and 512 would be expected if recrystallization were not occurring in warm rolling; however, the presence of beta phase in long stringers and along grain boundaries, rather than in a more uniform distribution, was not. The cause of this non-uniformity was thought to be variations in solidsolution magnesium content, with higher magnesium content

near grain boundaries, due to incomplete homogenization of the dendritic structure. Additional solution treatment at a higher temperature would be a way of reducing this.

Specimens 513 and 514 were processed exactly as 511 and 512, respectively, with the exception of solution treatment. After their initial solution treatment at 440°C for 20 hours, an additional solution treatment of 20 hours at 490°C was given. Examination of the final microstructure revealed no significant differences between them and 511 and 512, however. Tensile properties were slightly different; ductility was slightly increased and strength slightly decreased. The conclusion reached was that a gradient of magnesium content either did not exist or was not remedied by the additional solution treatment, or was not the cause of the grain boundary precipitate. The engineering stressstrain curve for specimen 514 is shown in Figure 13.

G. EFFECTS OF NON- ISOTHERMAL PROCESSING

At this point, full recrystallization had still not been induced under any conditions utilized. High strength with good ductility had been achieved, but a homogeneous microstructure had not. Following the approach of Sherby [Ref. 4], a series of non-isothermal processing experiments were conducted on both 51 and 54 series alloys. Because of the need for rapid rolling, the standard 5%-10% rolling schedule was changed to a fixed reduction of 0.040 inch (1 mm) per pass to facilitate rapid resetting of the rolls. This does not

produce any major change in the pattern of strain rate during rolling, as can be seen in Figure 8.

Specimens 517, 517WR, 518, 519, 542 and 543 were rolled, at least partially, as temperature was falling from 440° C. Specimens 544 and 545 were both worked isothermally, first at 440°C, then at 300°C. The details of these processes can be found in Table IV. The net result of these non-isothermal processes was to produce higher strength but less ductile materials in the 51 series alloys (10.2% Mg only) and lower strength, more ductile materials in the 54 series alloys (0.5% in addition to 10.2% Mg) as illustrated by Figures 15 and 16. Microstructurally no significant changes relative to isothermally rolled materials were found in samples taken from finished materials; the grain structure remained banded and unrecrystallized. Analysis of end pieces of the 54 series material, cut from the rolling billets in mid process, produced the first clue that higher strains were necessary to induce recrystallization. These end pieces contained material much more highly strained than material from the center of the finished material due to local folding over of the billet ends. Typically, microscopy samples were taken from the central regions of the rolled plate. In the highly strained regions some recrystallization had taken place.

H. ROLLING TEMPERATURE EFFECTS

The thermomechanical processes used in this series of experiments were isothermal. The specimens involved were

Fig. 15. Engineering stress strain curves for the 10.2% Magnesium Alloy. Each sample has a different thermomechanical process. Note that the four non-isothermal processes all produced a higher strength, lower ductility alloy than the isothermal process.

512, 516 and 5111. The processing of 512 was extensively discussed earlier; 516 was prepared and rolled in exactly the same manner, except that the isothermal rolling temperature was 340° C; 5111 was not air cooled after solution treatment; rather, it was directly rolled at 440°C. A minor change was made in its rolling schedule to lower the average strain rate. Figure ⁸ shows the average strain rate which resulted from the rolling schedule used. Table IV presents a more complete description of the processes used for this experiment. Figure 17 depicts the engineering stress-strain curves obtained from tensile testing these series of specimens. Most notable is the difference between the curve for 5111 and those of 512 and 516. The very large elongation, 33.3% at room temperature, is unmatched in aluminum alloys of this strength, 54 KSI (441 Mpa).

The microstructure of specimen 512 has already been discussed (Figure 10) . The microstructures of specimens 516 and 5111 are shown in Figure 18. Because of the striking contrast in microstructure, it is difficult to comprehend that they are of the same material. Specimen 516 is very similar to 512; banded, with stringers of beta phase primarily along elongated grain boundaries. Specimen 5111 grains are equiaxed and no precipitates are seen in either the grain interiors or in the grain boundaries. Clearly, recrystallization has occurred. The benefits of a uniform, equiaxed grain size and solid solution hardening are fully realized here.

This figure demonstrates the effect of rolling temperature Fig. 17. on engineering stress-strain curves. All specimens are of the same composition and received essentially the same thermomechanical process with the exception of rolling Sample 5111 has a fully recrystallized microtemperature. structure.

 (b)

(c)

(d)

Fig-. 18. Micrographs (a) and (b) are cross-sectional and longitudinal views of sample 516; (c) and (d) are the same views of 5111. The dramatic change in microstructure upon \bar{z} recrystallization. Micrographs are at 100X prepared by method 2.

I. PROCESS REFINEMENTS

From specimen 5111, the knowledge of a way to achieve recrystallization had been acquired. The grain size in that material, however, was larger than optimum; also, it was processed in the region of solid solubility and thus no benefit is obtained from dispersion hardening via precipitated beta.

Refinement of the grain size became the object of the next experiment. Samples of the rolling billet used for 5111 had been taken at a true strain of about 0.9. Micrographs of the structure at this point (Figure 19) , show that partial recrystallization has occurred. Where recrystallization has occurred the grains are equiaxed and smaller than those of the finished material. Unrecrystallized areas remain, similar in structure to the initial, as-cast dendritic structure. Also to be noted at this point is the absence of precipitates, either in the grain boundaries or grains interiors. Apparently, the necessary misorientation of a subgrain structure for recrystallization, induced via straining, was reached in the higher-strain areas at an average true strain of less than 0.9, and at some higher strain value, between 0.9 and 2.0, in low-strain areas. In the interval between partial and complete recrystallization, those grains formed first had grown to the size shown in 5111 in its finished form.

The isothermal rolling of 51 series specimens at 300°C had been accomplished easily. At 440°C, rolling proved more

(b)

Fig-. 19. Micrograph (a) shows a portion of specimen 5111 after true strain of 0.9 in cross section where complete recrystallization has occurred; (b) is of the same sample and illustrates the difference in grain size between recrystallized and unrecrystallized grains. 100X, prepared by method 2

difficult due to the development of hot shortness in the alloy. These two temperatures also represent areas where recrystallization had and had not occurred. Grain growth being dependent on time at 440°C, the next series of experiments were to determine if 44 0°C was the minimum recrystallization temperature for this alloy, and if a true strain of 1.0 was sufficient for complete recrystallization. The solvus temperature for this alloy is 380°C; therefore, a temperature above this, 400°C, was selected. A billet was given a standard solution treatment of 20 hours at 440°C and air cooled; it was then reheated to 400°C and rolled isothermally to a true strain of 1.0 and then air cooled. At this point the billet was cut into two equal parts and a sample taken. Figure 20 shows that complete recrystallization had occurred, but grains were not uniform in size and were somewhat elongated. In addition, considerable precipitate was present, some within grains but mostly in grain boundaries. The two halves of the billet were designated 5112 and 5113. Specimen 5112 was reheated and rolled to completion at 400°C; 5113 was solution treated for 20 hours at 400°C, air-cooled, then reheated and rolled to completion. Based on microscopy (Figures 21 and 22) , the following conclusions were reached: first, the grains grew and became more elongated without any substantive change in beta distribution; second, the additional solution treatment of 5113 did not resolution the beta phase to a significant

(b)

Fig. 20. These micrographs are cross sections of specimen 5112 at a true strain of 1.0. (a) clearly shows recrystallization has occurred, and (b) illustrates that precipitates are mostly in the grain boundaries . Both micrographs show the grains are slightly elongated and not of uniform size, (a) $100\overline{x}$, (b) $400\overline{x}$, prepared by method 2.

Fig. 21. Specimens 5112, (a) and (b), and 5113 (c) and (d) in their finished condition. (a) and (b) are cross sections, (b) and (d) are longitudinal views. Contrast this micrograph with those at a true strain of 1.0 to see the microstructural changes, 100X, prepared by method 2.

(b)

Fig. 22. Specimen 5113 after an additional solution treatment of 20 hours at 400°C at a true strain of 1.0. Grains are more uniform and larger, and the grain boundary precipitates remain. (a) 100X, (b) 400X, prepared by method 2.

amount; third, further recrystallization did not occur during the processing after division of the original billet. Tensile testing of specimens 5112 and 5113 produced engineering stress strain curves very similar in form to that of 5111. Strength improved about ⁷ KSI and elongation decreased about 7%.

The final experiment with this alloy had the same immediate objective as the one just previous. As was noted earlier no recrystallization occurred during isothermal processing at 300°C and rolling was easy, i.e. there were no problems with cracking, at this temperature. Because recrystallization had been found to be strain and temperature dependent and because grain growth rapidly occurred within the temperature range required for recrystallization, the attempt here was to first satisfy the strain requirement then the temperature requirement. This would allow for less time in the grain growth temperature region and result in a smaller grain size.

A single billet was given the standard solution treatment, then air cooled. Subsequently it was reheated to 300°C and isothermally rolled for 20 passes, being reduced .040 inch (10 mm) per pass, to a true strain of 1.10 (a thickness of approximately .4 inches (1.0 cm)). At this point the sample was air cooled, and divided into two equal parts designated 5114 and 5115. From this point, reduction to final thickness, using the same rolling schedule, requires only six passes. Each specimen was rehated and rolled isothermally to completion

in a period of approximately one hour. The rolling temperature was 440°C for 5114 and 420°C for 5115.

Micrographs of the resulting structures are shown in Figure 23. Examination of these micrographs demonstrate the following: first, the lower rolling temperatures produced a finer and elongated grain structure; second, grain boundary precipitates are present in 5115 but not in 5114; third, the 300°C processing prior to elevated processing did trigger complete recrystallization. Comparison of these micrographs with those of 5112 and 5113 shows that precipitates decreased with increased rolling temperature, being more pronounced in the grain boundaries of 5112 and 5113, reduced in 5115 and absent in 5114.

Tensile testing on these materials was conducted and the results are listed in Table IV, and graphically illustrated in Figure 24. Several comparisons with previous experiments are noteworthy. First are grain size effects; the microstructural difference between 5111 and 5114 seem to consist only of grain size, the smaller grain size adding about ⁵ KSI in strength. Second, strain-induced precipitation dominates the solutioning reaction at finishing temperature of 420°C and below; third, the more nearly equiaxed grain structure of materials final-processed at 440°C indicates that at this temperature the recrystallization process is rapid.

 (c) (d)

Fig. 23. These micrographs illustrate the differences in microstructure for rolling temperatures of $440\degree$ C (a) and (b); and $420\degree$ C (c) and (d). (a) and (c) are cross-sectional views, and (b) and (d) are longitudinal. 100X, prepared by method 2.

Fig. 24. Engineering stress strain curves for specimens ⁵¹¹⁴ and These curves illustrate the change in tensile properties due to rolling temperature. 5114 was finished at 440°C and 5115 was finished at 420°C.

VII . CONCLUSIONS AND RECOMMENDATIONS

The tensile properties developed in this study by warm thermomechanical processing are superior to those of non-heat treatable aluminum alloys commercially available. Further improvements in tensile properties are to be expected, based on a better understanding of the mechanisms involved in microstructured refinement during thermomechanical processing. Many of the problems encountered in previous work in this area have been solved and others have been bounded.

Future studies of high strength aluminum magnesium alloys should direct their efforts at the following areas:

1. Refinement of the recrystallization process is needed. This thesis has concentrated on this phenomena in 7% Mg and 10% Mg alloys. Investigation in higher magnesium-content alloys is needed if maximum benefit is to be gained from decreased density due to the Mg content.

2. The alloy containing .5% copper exhibited improved strength over the plain ten percent magnesium material. Investigation into its role in recrystallization may allow for still further improvement in microstructural control.

3. Additional research into the mechanism of straininduced precipitation is needed to refine the processing procedure so as to obtain the optimum in volume, shape and distribution of second phase.

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