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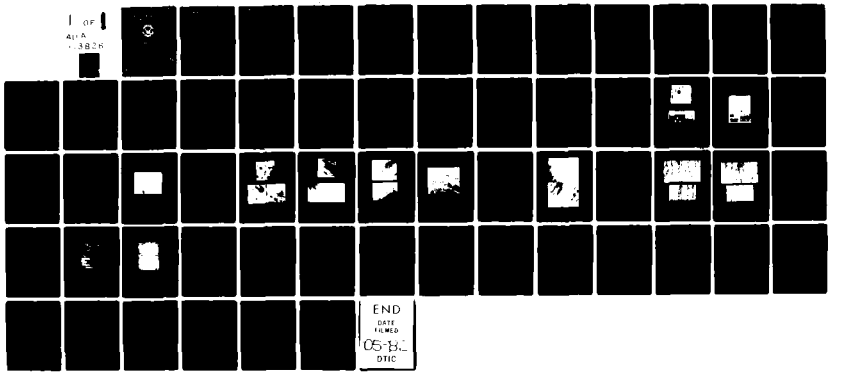

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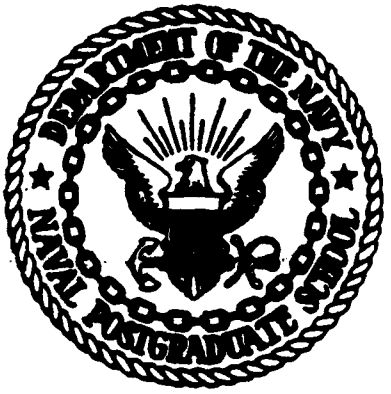

THESIS

THE INFLUENCE OF SOLUTION TREATMENT TIME AND QUENCH RATE ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HIGH-MAGNESIUM ALUMINUM-MAGNESIUM ALLOYS

by

Reuben H. Shirah

December 1981

Thesis Advisor: T. R. McNelley

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Aluminum alloys containing 8-10 wt pct magnesium with further minor additions of Cu and Mn were solution treated for either 10 hours or 24 hours and subsequently quenched in either oil or water. Microstructural analysis by transmission electron microscopy and optical microscopy demonstrated that a 24-hour solution treatment when followed by water quenching was effective in retention of Mg in solution promotiong more uniform precipitation

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during subsequent warm rolling, especially in an alloy containing 8.14 wt pct Mg-0.41 wt pct Cu. Tension and fatigue testing revealed improved fatigue resistance for this alloy when comparison was made to shorter solution treatment times and less severe quench conditions. The addition of Mn, possibly in conjunction with increased solution treatment temperature, appears to be especially effective in development of a homogeneous microstructure; an alloy containing 10.2 wt pct Mg-0.52 wt pct Mn exhibited a yield strength of 398 Mpa (57.8 Ksi), a tensile strength of 455 Mpa (75.8 Ksi) with 11 pct elongation to fracture.

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The Influence of Solution Treatment Time and Quench Rate on Microstructure and Mechanical Properties of High-Magnesium Aluminum-Magnesium Alloys

by

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ABSTRACT

Aluminum alloys containing 8-10 wt pct magnesium with further minor additions of Cu and Mn were solution treated for either 10 hours or 24 hours and subsequently quenched in either oil or water. Microstructural analysis by transmission electron microscopy and optical microscopy demonstrated that a 24-hour solution treatment when followed by water quenching was effective in retention of Mg in solution promoting more uniform precipitation during subsequent warm rolling, especially in an alloy containing 8.14 wt pct Mg-0.41 wt pct Cu. Tension and fatigue testing revealed improved fatigue resistance for this alloy when comparison was made to shorter solution treatment times and less severe quench conditions. The addition of Mn, possibly in conjunction with increased solution treatment temperature, appears to be especially effective in development of a homogeneous microstructure; an alloy containing 10.2 wt pct Mg-0.52 wt pct Mn exhibited a yield strength of 398 Mpa (57.8 Ksi), a tensile strength of 455 Mpa (75.8 Ksi) with 11 pct elongation to fracture.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

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I. INTRODUCTION

The purpose of this thesis was to investigate the response of high-magnesium aluminum-magnesium alloys to solution treatment time and subsequent quench rate prior to a warm rolling process. Particular emphasis was given to microstructural homogeneity and refinement, especially of the intermetallic precipitate. Also studied were tensile mechanical properties and fatigue characteristics of several thermomechanically processed alloys.

This research extends the knowledge obtained in previous work [Ref. 1-8] and in particular follows closely Johnson's [Ref. 7] work and Cadwell's research [Ref. 8]. Transmission Electron Microscopy (TEM), optical metallography, and tensile and fatigue testing were employed to determine the effects on the alloys of increased solution treatment time and also the effect of quench rate on microstructural changes. The three aluminum-magnesium alloys investigated were Al-8.14%Mg-0.41Cu, Al-10.2%Mg and Al-10.2%Mg-0.52%Mn.

II. BACKGROUND

A. ALUMINUM-MAGNESIUM ALLOYS

Aluminum and its alloys are second only to steel in the metals industry [Ref. 9]. The use of aluminum alloys is likely to grow especially considering the current emphasis on saving energy as these alloys offer high strength to weight ratios.

Aluminum-magnesium alloys are of particularly high interest in that magnesium addition results in alloys of lower density than even pure aluminum and of good ductility, moderate strength and good weldability. Due to the tendency for precipitation of magnesium from solid solution at grain boundaries, many of the commercially produced aluminum-magnesium alloys today contain less than five weight-percent magnesium. The aluminum end of the Al-Mg system on an equilibrium phase diagram (Fig. 1) shows a eutectic at approximately 450°C between aluminum containing 15.35% Mg in solid solution, the beta (β) phase, Al_8Mg_5 and a liquid. The solid solubility of Mg decreases to about 2.0%Mg at 25°C.

The basic concept of this work has been to achieve control of the beta phase precipitate. By holding these alloys at temperature within the single phase region until all of the magnesium is in solid solution and then rapidly quenching the material, the beta phase should stay in solution and not

precipitate out at the grain boundaries. Then, by using a warm rolling sequence the beta precipitate can precipitate in grain interiors as a uniform dispersion. The added strength of these aluminum-magnesium alloys can then be achieved by an increase in dislocation density due to warm rolling and the uniform precipitation. The effects of solution treatment time and quench rate have been recurring questions in previous work and are examined and discussed in this research.

The wrought alloys of the 5XXX series and the cast alloys of the 5XX.X series (all aluminum-magnesium alloys) have a high resistance to corrosion. Conversely, the precipitation of the $\beta(Al_8Mg_5)$ precipitate at grain boundaries in wrought Al-Mg alloys containing greater than 3%Mg makes these alloys susceptible to exfoiliation and stress corrosion cracking. Various tempers have been developed to produce microstructures having the precipitate finely dispersed within the grains [Ref. 10]. This work and previous work on high-magnesium aluminum-magnesium alloys also has been aimed at producing a homogeneous, uniformly dispersed β but at much higher Mg content than normally considered.

B. PREVIOUS WORK

Grandon [ref. 4] initiated work in the aluminum-magnesium alloys containing 7-12%Mg. He used a variety of solution treatment times (5-60 hours) on these alloys and concluded

that the β phase redissolved within five hours and little apparent gain was evident with increasing the solution treatment time beyond 20 hours. This conclusion was reached by annealing treatments with no mechanical working being applied. Other problems encountered included quench cracking and non-homogeneous microstructures. One recommendation made by him was to further explore hot working as part of the thermo-mechanical process to achieve more complete refinement and homogenization by recrystallization prior to or during warm rolling. His work demonstrated that high strengths were attainable but that greater attention be given to micro-structural homogeneity.

Johnson [Ref. 7] concluded that a 10-hour solution treatment provided sufficient time to redissolve all the magnesium in an aluminum matrix with hot working by upset forging to further promote homogenization during the solution treatment stage. He also standardized the solution treatment process to investigate the effects of warm rolling temperature on properties. He concluded that the 0.2% offset yield strength decreased and the ductility increased as the warm rolling temperature was increased. In his investigation of an alloy containing Al-10.2%Mg-0.52%Mn, he noted grain boundary precipitation in the oil-quenched samples and recommended this alloy only be water quenched to keep the precipitate out of the grain boundaries. The data from his research on the alloys also studied here is presented in Table I.

Cadwell [Ref. 8] examined the fatigue resistance of two alloys (Al-8.14%Mg-0.41%Cu and Al-10.2%Mg). Water and oil quenching were employed following a 10-hour solution treatment. He noted that the microstructure of the materials oil quenched after solution treatment was more coarse with more grain boundary precipitation than those alloys water quenched after solution treatment. He also observed a dense dislocation substructure in the 10.2%Mg binary alloy by Transmission Electron Microscopy (TEM) and concluded that the strengthing mechanism in the alloys appeared to be a dislocation substructure developed by warm rolling. To facilitate fatigue testing, Cadwell [Ref. 8] conducted tensile testing and his results are included in Table II.

Cadwell [Ref. 8] suggested more attention be given to the effects both of solution treatment time and subsequent quench rate as determined by quench medium to clarify questions raised in previous work in this area. Also, that a more detailed TEM investigation be conducted more clearly to define the microstructure, especially the dislocation substructure, in these alloys. These suggestions provide the basis for this current work.

III. EXPERIMENTAL PROCEDURES

A. MATERIAL PROCESSING

The alloys selected for study were obtained as direct-chill cast ingots 127mm (5 in) in diameter by 106mm (40 in) in length, that were produced by Alcoa Center for Technology, Pittsburgh, Penn. In manufacture, 99.99% pure Al was used with various alloy additions as listed below [Ref. 7]:

Alloy #	Serial #	Si	Fe	Cu	Mg	Ti	Be	Mn
1	501303A	0.01	0.03	0.40	8.14	0.01	0.0002	0.00
2	501299A	0.01	0.03	0.00	10.2	0.01	0.0002	0.00
3	501300A	0.01	0.03	0.00	10.2	0.01	0.0002	0.00

The as-received ingots were sectioned into billets of 96mm (3.75 in) length by 32mm (1.25 in) by 32mm (1.25mm) in cross-section to facilitate thermomechanical processing.

The thermomechanical processing was completed using three different schemes: (1) 10 hour solution treatment followed by an oil quench, as given by Johnson [Ref. 7] and also used by Cadwell [Ref. 8]; (2) 24 hour solution treatment, followed by a water quench; and (3) 24 hour solution treatment followed by an oil quench.

Under scheme (1) the billets were solution treated for nine hours at 440^{o}C, isothermally forged to break up the dendritic, as cast structure, and then sclution treated an

additional hour before oil quenching. Under schemes (2) and (3) the billets were solution treated for 23 hours at 440°C, isothermally forged, then solution treated for an additional hour, followed by either an oil or water quench. One billet of alloy #3 (the alloy containing 0.52%Mn) was thermomechanically processed using scheme (2) except that the solution treatment temperature used was 490°C. This higher temperature was necessary to dissolve a phase, likely $MnAl_6$, present in the as-cast condition [Ref. 11]. Isothermal upset forging (hot working) of the billets was conducted to reduce the maximum dimension of the billets to a thickness of approximately 32mm (1.25 in).

The forged billets were further processed following the warm rolling procedure developed by Johnson [Ref. 7]. A warm rolling temperature of 300°C was selected to facilitate comparison with Johnson's work [Ref. 7] on tensile mechanical properties and comparison with fatigue characteristics reported by Cadwell [Ref. 8]. The warm rolling was accomplished by first preheating the billets for 10 min prior to the first pass at 300°C. Rolling was then accomplished at 0.8mm/pass (0.030 in/pass) and with a reheating time of 2 minutes between passes. An average temperature drop of 20 C° per pass(since the mill rollers are not heated) was reported by Grandon [Ref. 4]. Each pass through the mill requires approximately 15 sec. To maintain straightness

of the billets as they were rolled, a four-pass sequence was utilized [Ref. 7]. The rolling mill used is shown in Figure 2.

The billets were rolled to a final thickness of 4.8 - 5.1mm (0.190 - 0.200 in) to facilitate preparation of TEM samples from the short transverse plane (plane of material normal to the rolling direction). Tensile samples also were mechined from these rolled materials. Several billets were rolled to a thickness of 4.1 - 4.3mm (0.160 - 0.170 in) for machining into fatigue samples. It should be noted that these final thicknesses are greater than the final as-rolled thickness used in Johnson's work [Ref. 7] and also in Cadwell's work [Ref. 8].

B. TRANSMISSION ELECTRON MICROSCOPY (TEM)

1. General Description

Thin foils for TEM analysis were prepared from these alloys representing the different solution treatment times and quench rates previously described. Foils generally were prepared with the foil normal parallel either to the rolling direction or the long transverse direction (the plane of the foil was parallel to the short transverse direction).

Several thin sections, 0.30mm (0.012 in) in thickness by 19.1mm (0.75 in) in length by 4.8mm (0.190 in) in width (i.e., the as-rolled thickness), were sectioned using an ISOMET Low-Speed Saw shown in Figure 2(b). These thin sections subsequently were electrolytically thinned to approximately 0.1mm (0.003 in) thickness using the Tenupol Thinning

apparatus shown in Figure 3. Two 3mm (0.118 in) discs were mechanically punched from each sample for further thinning to penetration for TEM analysis.

2. Electrolytic Thinning

The electrolyte used was a mixture of 700ml methanol, 200ml glycerol and 100ml of perchloric acid. The concentration of perchloric acid is lower than that called for by Ref. 12, but the perchloric concentration can be increased as desired. The chemical composition used produced a suitable polishing for TEM analysis. Electrolytic thinning was accomplished using various currents, voltages, flow rates and temperatures. The optimum results were obtained using a current of 1.15-1.35 amps (controlled by sample dimensions and electrolyte temperature) at 20 volts and with maximum flow rate (10 setting on dial scale), and at a temperature between $5^{o}C$-$22^{o}C$. Although minor variations in thickness were observed in the thinned samples, the above technique produced material between 0.03-0.08mm (0.001-0.003 in) in thickness. Two important observations should be noted in using this thinning technique: (1) perchloric acid is highly reactive and leaves a powdery residue when it dries which is explosive, and (2) using perchloric acid when polishing aluminum does not result in a plateau for current density verses voltage, but rather results in a linear relation as shown in Figure 2 of Ref. 13.

Specimens to be thinned were first placed in the 10mm holders and placed in the Tenupol unit. Voltage, flow rate

and temperature conditions as described above were established. Approximately three minutes were required to thin the sample to 0.080mm (0.003 in). Thin foil discs, 3mm in diameter, were then punched out and placed in the 3mm holder for further thinning and penetration of the disc. The flow rate was held at 10 for these discs, but the voltage was lowered to 5-7 volts, reducing the current to 40-60 milliamps. A photocell stopped the pump and current flow in the Tenupol unit when penetration was made.

Specimen cleanliness is very important. The specimen holder was immediately rinsed in acetone at penetration of the disc. The disc was removed and rinsed again with acetone and then dried using an electric blow dryer. The specimens were placed in a dessicator to minimize contamination. The specimens were observed in the TEM within 12-15 hours after penetration. This procedure generally resulted in a good thin foil, although some artifacts were noted. Artifacts were more notable as conditions varied from those described. The Tenupol unit and associated equipment is shown in Figure 3.

C. METALLOGRAPHY

Samples for optical microscopy were mounted in bakelite and polishing was accomplished utilizing wet sand papers of 400 and 600 grit. Final polishing was completed using a magnesium oxide (Magomet) slurry. This slurry was put on a polishing wheel (Automet) and distilled water was supplied

by a controlled drip onto the wheel. The magnesium oxide slurry was replenished approximately every 15 seconds. Fast speed (1150 RPM) was utilized on the Automet Polisher with approximately 5-10 pounds of pressure.

The mounted samples were etched with Keller's reagent [Ref. 13] for 40 seconds and examination was accomplished utilizing a Zeiss optical microscope. Samples of each alloy also were electrolytically polished using the thinning procedure previously presented. These samples were electrolytically etched utilizing Barker's reagent [Ref. 13] and examined utilizing a Zeiss optical microscope with polarized light.

D. TENSILE TESTING

Tensile test samples were machined from each alloy from the as-rolled sections. The testing was accomplished utilizing an Instron Model TT-D Floor Model testing machine with a crosshead speed of 5mm (0.2 in) per minute and load-elongation data was autographically recorded. The tensile specimens were machined to have a 38mm (1.5 in) gage length. Details of the specimen design are illustrated in Figure 4.

E. FATIGUE TESTING

The specimen design and dimensions utilized is depicted in Figure 5 and is that designed and used by Cadwell [Ref. 8] in previous work. Specimens of each alloy were fatigue tested utilizing a Tatnall-Krouse Variable Speed Plate Fatigue Testing Machine.

Each specimen was clamped into an adjustable vise, and the proper amount of weight as calculated by equation (1) below was applied to the free end to give the desired deflection for a desired stress amplitude:

$$P = \frac{Sbt^2}{6L} \qquad (1)$$

where P is the applied load, S is the desired stress, b = 0.531 in for all specimens, L = 3.00 in for all specimens and t was approximately 0.1 in $\pm$ 0.05 in. A detailed analysis of the deflection desired, specimen geometry and flexure analysis was presented by Cadwell [Ref. 8].

Each sample was loaded to the desired stress amplitude and tested through fully reversed bending at 1980 cycles/minute. The fatigue testing machine counts the fully reversed bending cycles and has an automatic cut-off switch which stopped the machine when the specimen fractured.

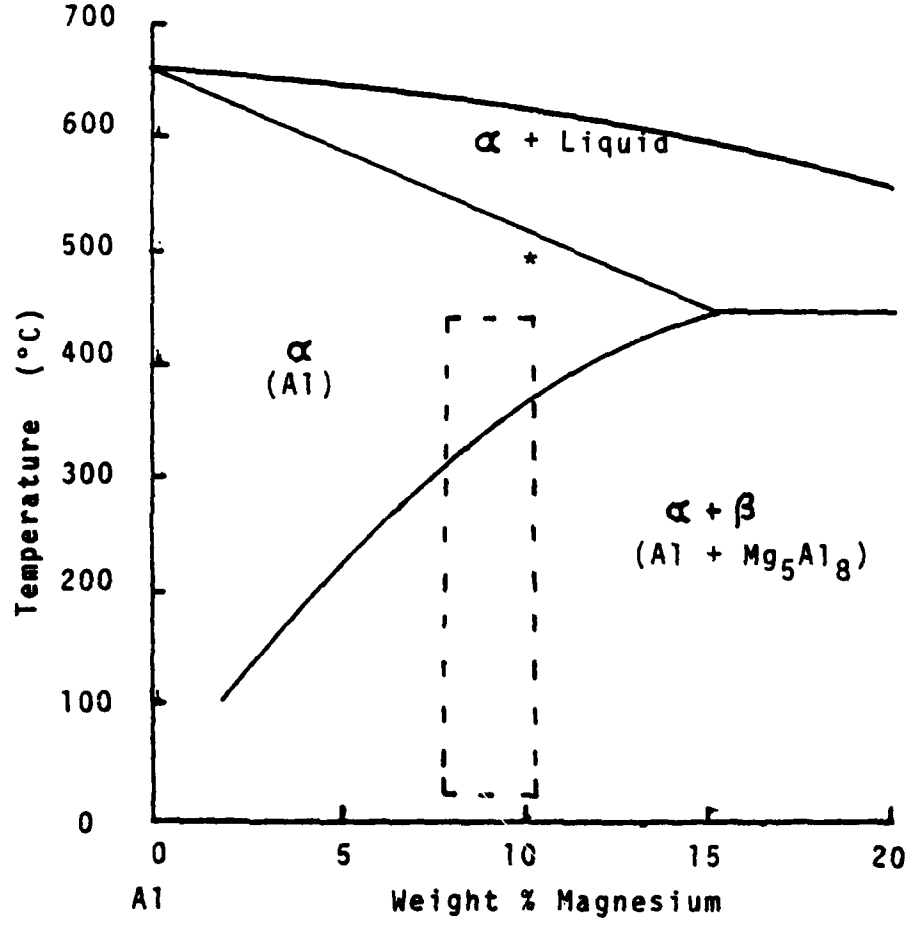
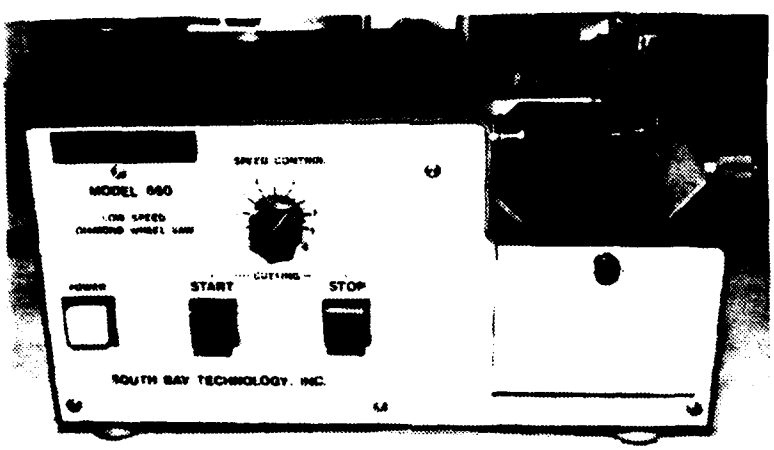


Figure 1. Partial Aluminum-Magnesium Phase Diagram. Dashed in area represents compositions used in this work. * Represents the solution treatment temperature used on the Al-10.2%Mg -0.52%Mn alloy.

(a)



(b)

Figure 2. (a) Fenn Variable-Speed Electric Drive Rolling Mill (b) South Bay Technology Model 650 Low Speed Diamond Saw

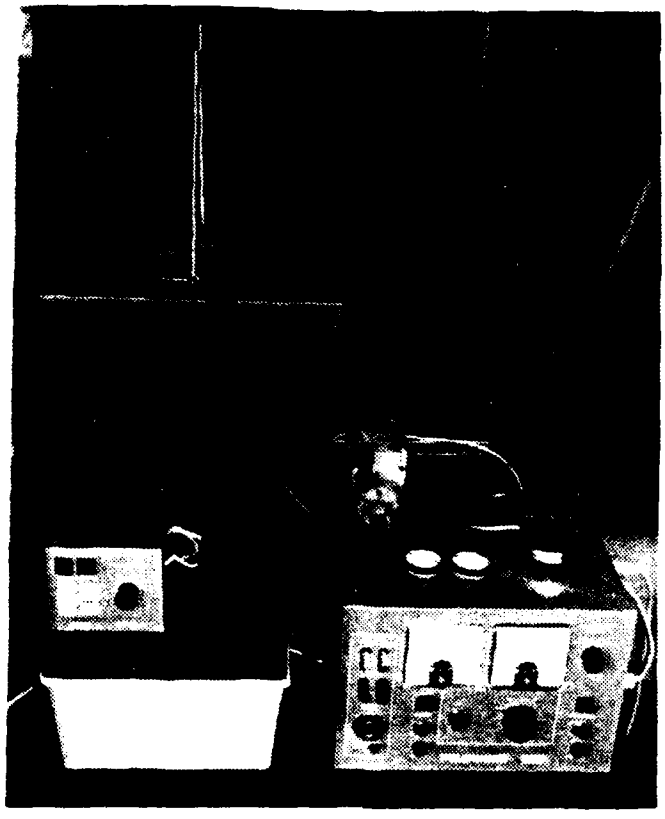


Figure 3. Tenupol Thinning Apparatus. Electrolytic Thinning unit on left, Polipower unit on right and Refrigeration unit in background.

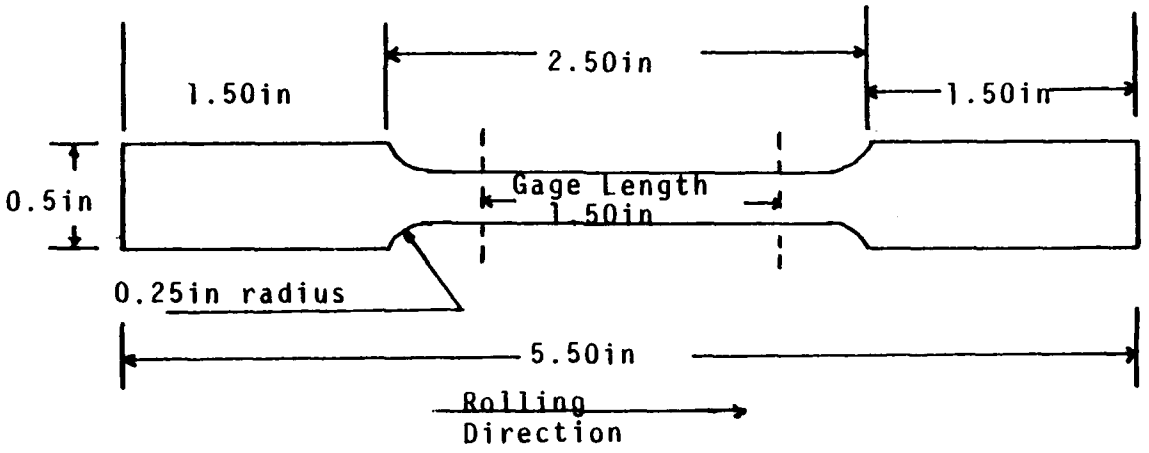


Figure 4. Tensile Test Specimen

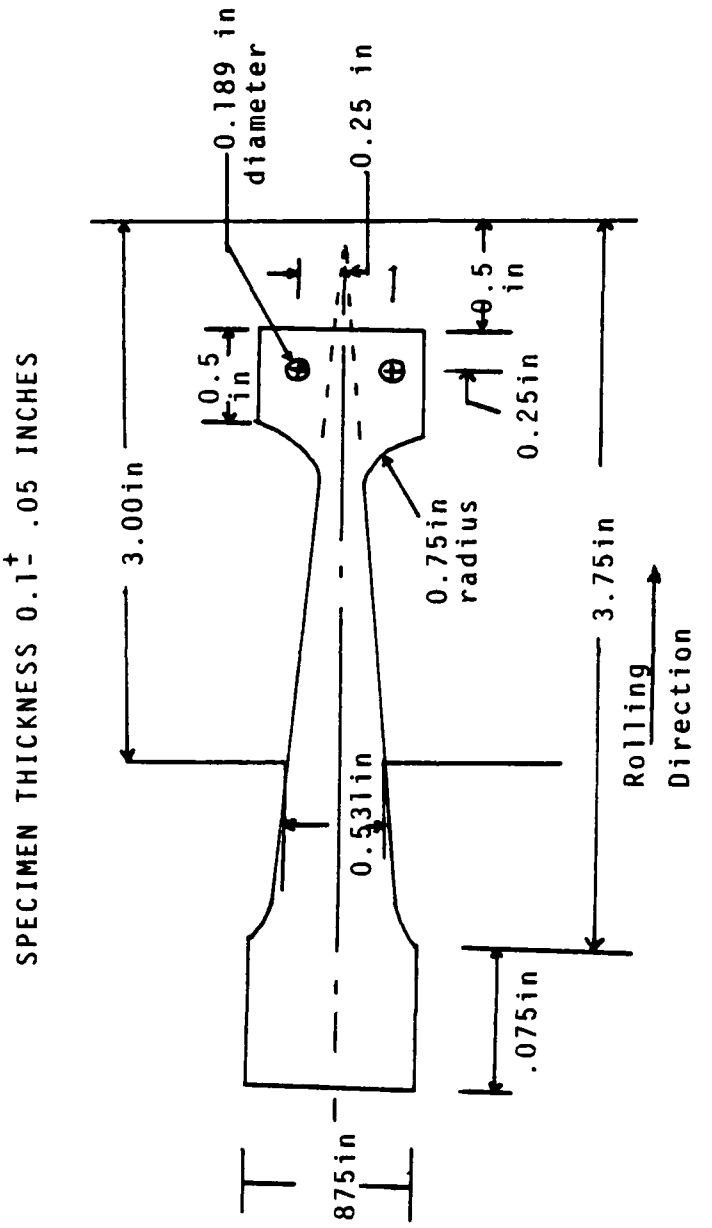


Figure 5. Fatigue Test Specimen

IV. RESULTS AND DISCUSSION

A. MATERIAL PROCESSING

A standardized thermomechanical processsing technique was utilized to process materials for this investigation. For the purposes of this study, the variables of interest were solution treatment time and subsequent quench rate. Variation in quench rate was achieved by using either oil or water as a quench medium. Cadwell [Ref. 8] reported severe cracking when the Al-8.14%Mg-0.41%Cu alloy was warm rolled at 0.040 in/pass. He subsequently used a rolling reduction of 0.020 in/pass without cracking problems. This same alloy was solution treated, water quenched and warm rolled in this research at a reduction of 0.030 in/pass and without cracking problems.

Billets of the Al-10.2%Mg-0.41%Cu alloy were solution treated, forged and either oil or water quenched in this study and severe cracking was observed upon quenching. Mondolfo [Ref. 15] indicates that at 300°C it is possible to have a ternary phase of composition $CuMg_4Al_6$ if sufficient magnesium is present. The solid solubility of copper in aluminum also decreases as the magnesium additions increase in the range of 7-15% magnesium. This ternary phase precipitates in grain boundaries. These factors may be responsible for the intergranular cracking observed in this alloy during the course of this research (Fig. 6). In particular, the observation of increased susceptibility in the higher Mg alloy supports this view.

Figure 6. Intergranular cracking of the Al-10.2%Mg-0.41%Cu alloy, during either oil or water quenching from a 24-hour solution treatment and forging @ 440°C. (100X)

The effect of increasing the solution treatment temperature to 490°C was examined for the Al-10.2%Mg alloy. A billet of this alloy was solution treated at 490°C for 15 hours; this billet previously had been forged after a 10-hour, 440°C solution treatment with subsequent oil quenching. Upon water quenching from 490°C, the billet immediately intergranularly cracked as a result of hot shortness. This is believed to be quench cracking as 490°C is close to the liquidus temperature in this alloy. Subsequent work, however, should address the question of solution treatment temperature as this area was given only limited examination here.

B. TRANSMISSION ELECTRON MICROSCOPY

Of particular interest in this research was the development of properly prepared thin foils for TEM analysis. For this reason the TEM work will be discussed first. Thin foils of each of the alloys were prepared from the as-rolled material with the foil normal parallel to the rolling direction. Aluminum-magnesium alloys present a challenge when preparing thin foils for TEM work. For example, the tendency for small precipitates to be dissolved out of the foil was noticed, therefore leaving a small hole before the foil was properly thinned. This and other artifacts were often noted in foils prepared from these warm-rolled alloys.

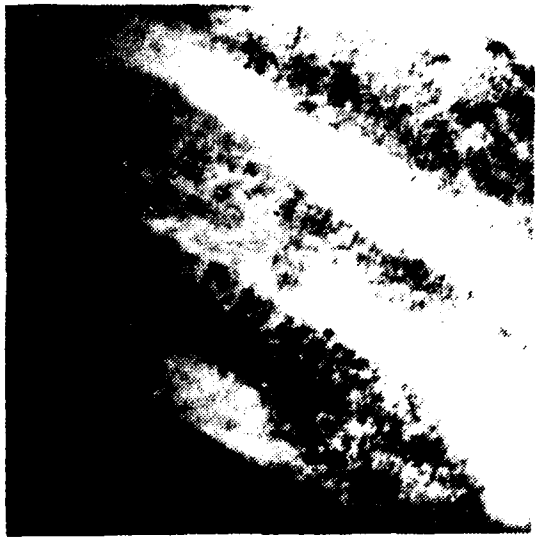
No clear indication of uniform precipitation or uniform fine dislocation substructure is seen in Figures 7 (a), (b),

(a) 1.0μm

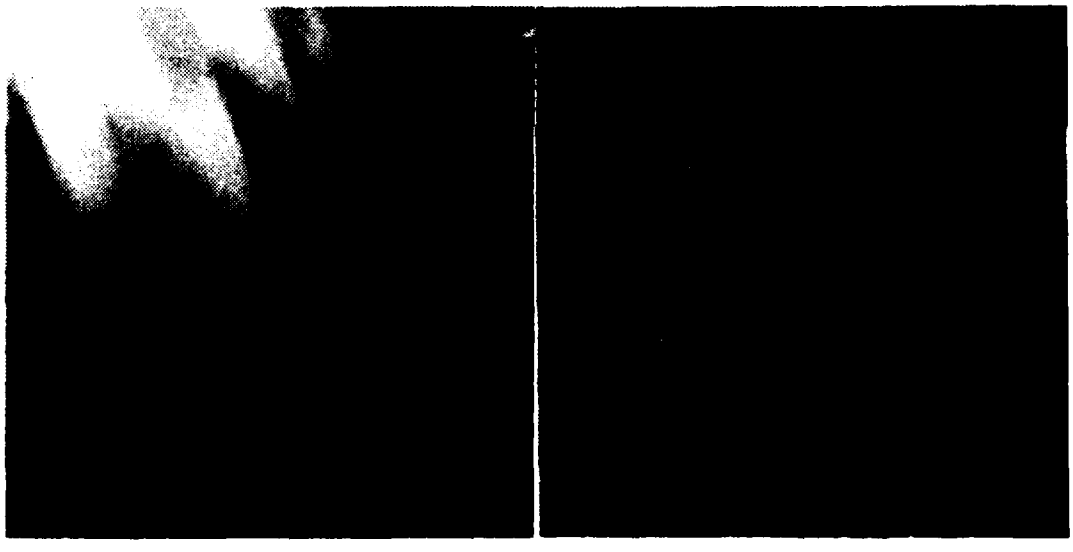
(b) 1.0μm

(c) 1.0μm

Figure 7. TEM micrographs from thin foils of Al-8.14%Mg -0.41%Cu solution treated @ 440°C, forged, quenched and warm rolled @ 300°C: (a) 10-hour solution treatment, oil quench, (b) 24-hour solution treatment, oil quench, (c) 24-hour solution treatment, water quench.



(a) .5μm



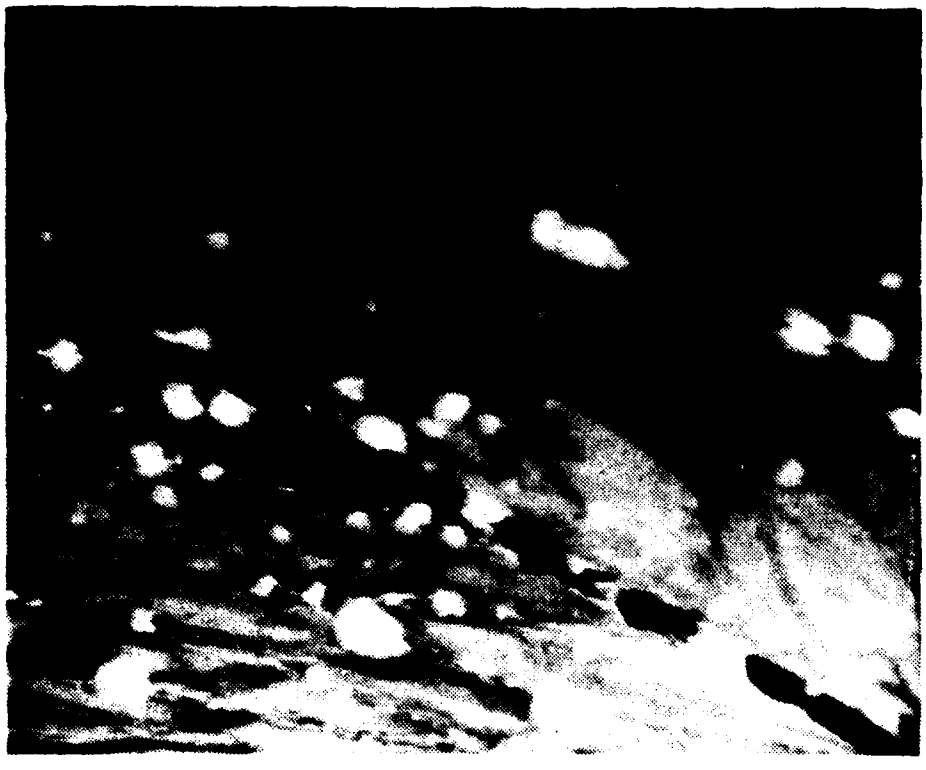
(b) .5μm (c) .5μm

Figure 8. TEM micrographs from thin foils of Al-10.2%Mg, solution treated @ 440°C, forged, quenched and warm rolled @ 300°C: (a) 10-hour solution treatment, oil quench, (b) 24-hour solution treatment, oil quench, (c) 24-hour solution treatment, water quench.

(a) 1.0μm

(b) 2.0μm

Figure 9. TEM micrographs from Al-10.2%Mg alloys, solution treated @ 440°C, forged, oil quenched and warm rolled @ 300°C: (a) 10-hour solution treatment, showing angular morphology of precipitates, (b) 24-hour solution treatment, showing areas where precipitates have been dissolved out of foil.



2.0μm

Figure 10. TEM micrograph of an Al-10.2%Mg solution treated 10 hours @ 440°C, forged, oil quenched and warm rolled @ 300°C showing long continuous precipitate in the grain boundary.

and (c) (a comparison of micrographs of Al-8.14%Mg-0.41%Cu from different solution treatment times and quench rates). Areas where precipitate particles likely existed are seen, but the precipitation is nonuniform. Some fine, nonuniformly distributed precipitate particles appear to have existed in the 24-hour solution treated, water quenched and warm rolled material (Fig. 7(c)) thin foil. Some fine substructure also appears in Figure 7(b).

Figures 8(a), (b) and (c) indicates a similar circumstance in the Al-10.2 wt pct Mg alloys. Figures 9(a) and (b) shows TEM micrographs from two Al-10.2 wt pct Mg alloys with 10 and 24-hour solution treatment times respectively. Figure 10 indicates the nonuniform, angular precipitate morphology previously noted by Cadwell [Ref. 8]. The existence of substructure in this figure is indicated by the precipitation of small particles on cell boundaries within the grain interior. Also this figure clearly indicates the existence of long, continuous precipitation in a grain boundary.

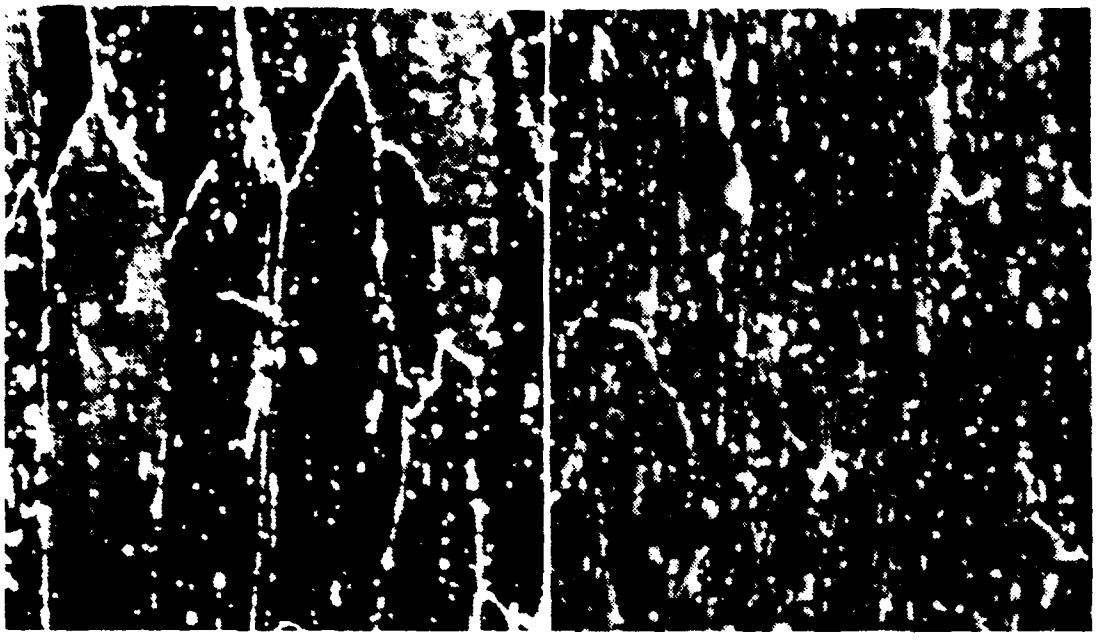
The Al-10.2 wt pct Mg-0.52 wt pct Mn alloy micrographs are shown in Figure 11. This alloy was solution treated for 24 hours at 490^{o}C and water quenched. This particular alloy illustrates a more uniformly distributed precipitate and a dislocation substructure is clearly evident. The precipitated second phase also seems to be in stringers and form

Figure 11. TEM micrographs from Al-10.2%Mg-0.52%Mn alloy, solution treated @ 440°C, forged, water quenched and warm rolled @ 300°C.

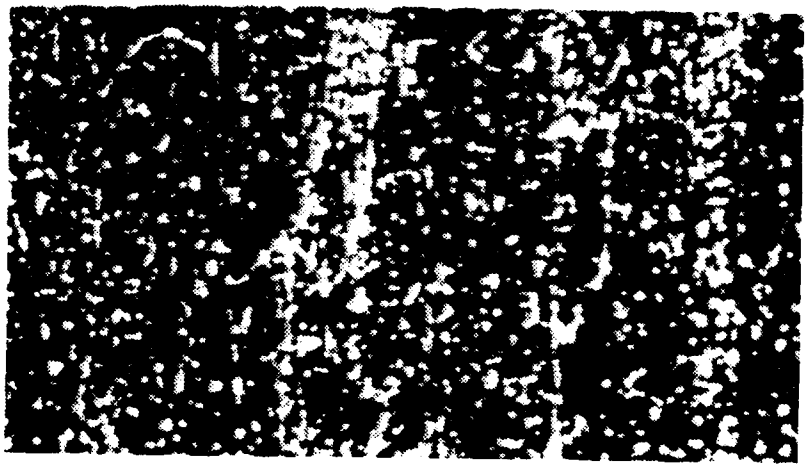
long, finely precipitated bands similar to the dispersion strengthened aluminum-magnesium alloys discussed in Ref. 15 but not as fine. The finer precipitates and substructure would account for the increased tensile strength and reduced ductility exhibited by this alloy as noted in Tables I and III; Table III summarizes the results of the mechanical testing of this research.

C. OPTICAL MICROSCOPY

Samples were electrolytically etched for optical microscopy and viewed using polarized light on a Zeiss optical microscope. Figure 12(a-c) are optical micrographs of the Al-8.14 wt pct. Mg-0.41 wt pct Cu alloy illustrating the effects of solution treatment time and quench rate on this material. These micrographs are of completely processed materials. The materials that were oil quenched prior to warm rolling show extensive grain boundary precipitation but with some improvement when solution treatment time is extended to 24 hours. In contrast, water quenching after 24 hours of solution treatment results in almost no grain boundary precipitation in this alloy. Figure 13(a-c) are optical micrographs of the Al-10.2 wt pct. Mg alloy illustrating the effects of solution treatment time and quench rate on this material. Extensive grain boundary precipitation is observed in the alloy solution treated for 10 hours only and oil quenched. Longer solution treatment improves homogeneity somewhat (Fig. 13(b)), however, a water



(a) (b)



(c)

Figure 12. Optical micrographs of Al-8.14%Mg-0.41%Cu solution treated, forged, quenched and warm rolled @ 300°C: (a) 10-hour solution treated, oil quench, (b) 24-hour solution treated, oil quench, (c) 24-hour solution treated, water quench. (300X)

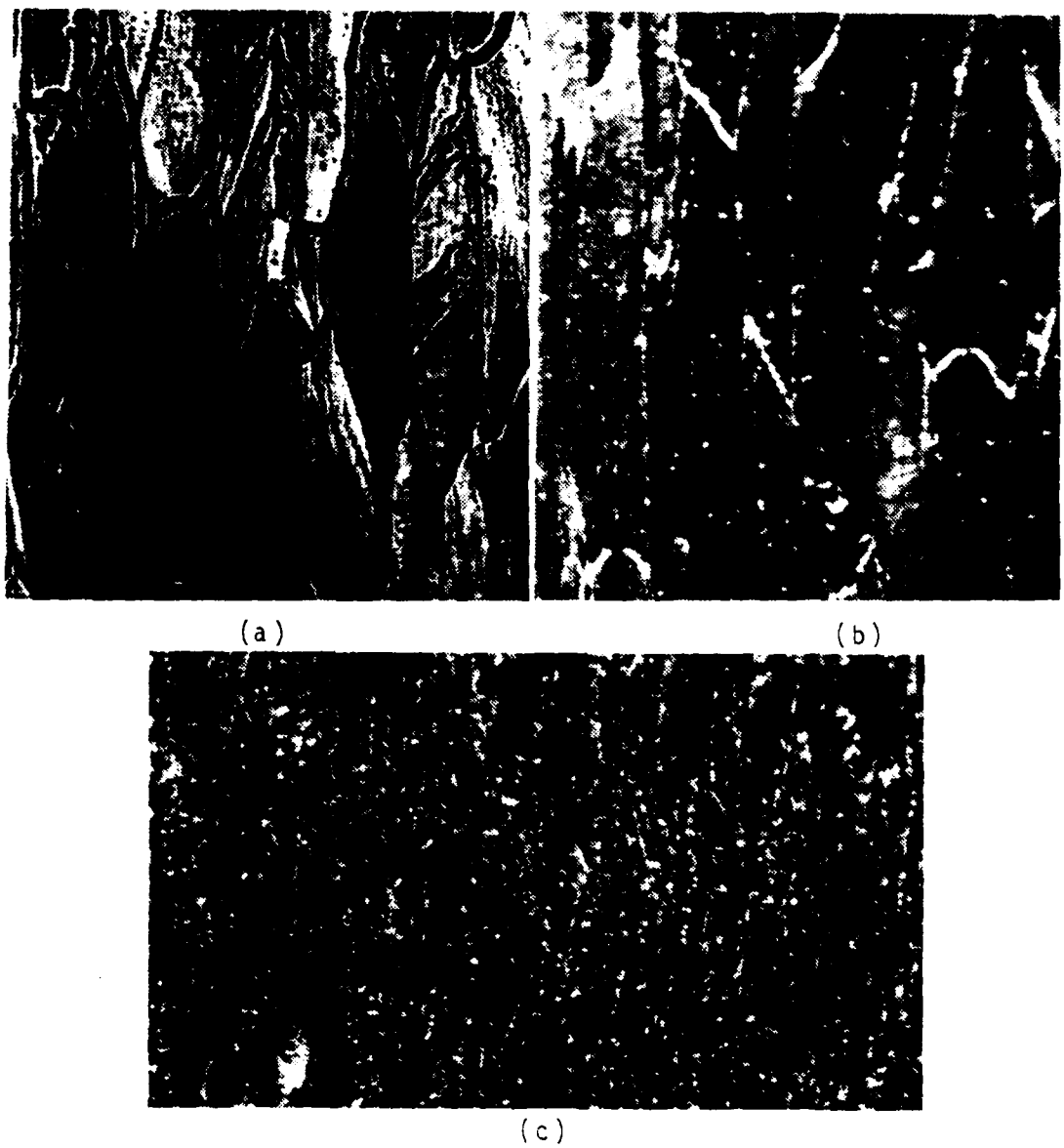


Figure 13. Optical micrographs of Al-10.2%Mg, solution treated @ 440°C, forged, quenched and warm rolled @ 300°C: (a) 10-hour solution treatment, oil quenched, (b) 24-hour solution treatment, oil quenched, (c) 24-hour solution treatment, water quenched. (300X)

quench after 24 hours solution treatment appears still more effective in retention of Mg in solution and in promoting more uniform precipitation in subsequent warm rolling (Fig. 13(c)). Upon warm rolling, the precipitates form in the interior of grains but precipitation still occurs in grain boundaries. In this alloy then, both solution treatment time and quench rate affect the resulting microstructure. Of the two variables, the quench medium appears more important in promoting uniform precipitation. In Figure 13(a), the grain boundaries appear to contain a continuous band of beta precipitates, while more of the precipitates in Figure 13(b) appear to have formed in the grain interiors, indicating a small effect due to more complete solutioning of this alloy from a 24-hour solution treatment. Figure 14, an optical micrograph of this alloy using nonpolarized light, illustrates more clearly the discontinuous precipitate pattern of the beta phase in grain boundaries.

The Al-10.2 wt pct Mg-0.52 wt pct Mn alloy shown in Figure 15 was initially solution treated at 490°C and then water quenched. This different procedure was necessary due to the presence of a third phase, as noted earlier. The microstructure of this alloy shows almost no evidence of grain boundary precipitation. It is homogeneous, with uniformly dispersed precipitation, and as noted earlier shows clear evidence for substructure formation. The effects of solution treatment time and quench medium were not investigated in this alloy and thus it is not possible to compare directly

to the previous two alloys discussed. In fact, these observations prompted the attempt to raise the solution treatment temperature for the Al-10.2 wt pct Mg alloy, which resulted in cracking during the subsequent water quenching of that alloy.

There are several questions raised in this study regarding the various effects of the solution treatment parameters. Extended solution treatment time and especially water quenching result in more uniform, homogeneous precipitation in subsequent warm rolling. These effects appear to be more pronounced in the alloy containing Cu and this is likely due to reduced diffusion rates for Mg when Cu is present [Ref. 11]. This cannot be stated unambiguously, however, as Mg content was not held constant. It should also be noted that more difficulties were encountered in rolling those alloys containing Cu.

The addition of Mn appears to have even greater effect in promotion refinement and homogenization. This, too, is subject to qualification in that the solution treatment temperature was necessarily different. This suggests, however, more detailed study of the Mn-containing alloy to evaluate solution treatment time, temperature and quench rate effects in it.

Figure 14. Optical micrograph of Al-10.2%Mg alloy solution treated 10 hours @ 440°C, forged oil quenched, and warm rolled @ 300°C showing discontinuous precipitates in the grain boundaries. (300X)

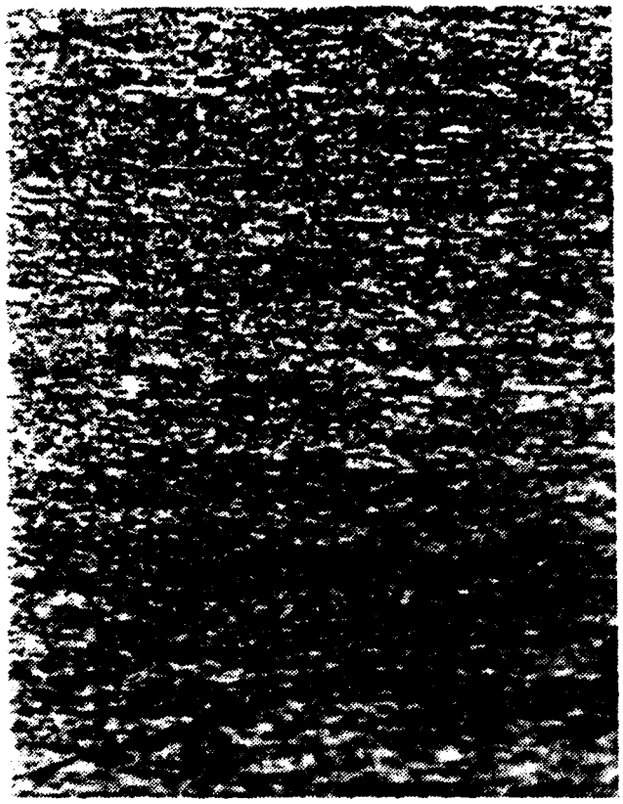


Figure 15. Optical micrograph of Al-10.2%Mg-0.52%Mn alloy, solution treated @ 490°C, forged, water quenched and warm rolled @ 300°C showing homogeneous microstructure. (300X)

D. TENSILE MECHANICAL PROPERTIES

Tension testing was conducted on the alloys of this research to provide data for analysis of the effects of processing variations and to compare with the data of Johnson [Ref. 7] and Cadwell [Ref. 8]. The data of Johnson [Ref. 7] and Cadwell [Ref. 8] are reported in Tables I and II while the data of this work appears in Table III. Examination of the data reveals that the yield and ultimate strengths obtained in this work are somewhat lower than those reported by Johnson [Ref. 7] while ductilities are considerably higher. Most of the differences noted, likely are due to the fact that the warm rolling in this work was terminated at a greater final thickness (to facilitate the TEM work). Hence, the warm rolling strain is less and lower strengths and higher ductilities result.

The data also suggest the water quenched materials to be somewhat higher in ultimate tensile strength but of similar ductility. This observation is somewhat different than that of Cadwell [Ref. 8] who reported lower ductility for the oil-quenched condition for the Al-8.14 wt pct Mg-0.40 wt pct Cu alloy. As in previous work, serrated yielding was apparent in the Al-10.2 wt pct Mg alloy and the Al-8.14 wt pct Mg-0.41 wt pct Cu alloy. This was observed to commence after a tensile strain of between 0.04 and 0.08 prior to which flow was smooth. Necking prior to fracture was observed in all cases.

The data obtained on the Al-10.2 wt pct Mg-0.52 wt pct Mn show it to be the highest strength alloy of those investigated,

consistent with the results of Johnson [Ref. 7]. Again, the data of this work suggest somewhat lower strength and greater ductility than obtained by Johnson [Ref. 7]. The mechanical properties obtained with this alloy, especially in light of the uniformity of the microstructure, are very attractive. The yield and ultimate tensile strengths obtained 398 Mpa (57 Ksi) and 522 Mpa (75 Ksi), respectively, with 11 percent elongation to fracture, compare favorably with property data for high strength alloys such as 7075. As noted previously, this alloy should receive greater attention in future work to develop more complete understanding of its properties and microstructure.

E. FATIGUE CHARACTERISTICS

Fatigue data was obtained on two of the alloys of this research, the Al-10.2%Mg alloy and the Al-8.14%Mg-0.41%Cu alloy. Data was obtained on materials solution treated for 24 hours and then either oil or water quenched. Materials solution treated only 10 hours were not included in this study in light of work on this condition by Cadwell [Ref. 8]. Direct comparison with that previous effort is possible since processing and test conditions were identical except for the different solution treatment times utilized. The data are presented in Figures 16-19, as stress-amplitude versus cycles-to-failure plots.

Figures 16 and 17 summarize results for the Al-8.14%Mg -0.41%Cu alloy for the oil and water quenched conditions,

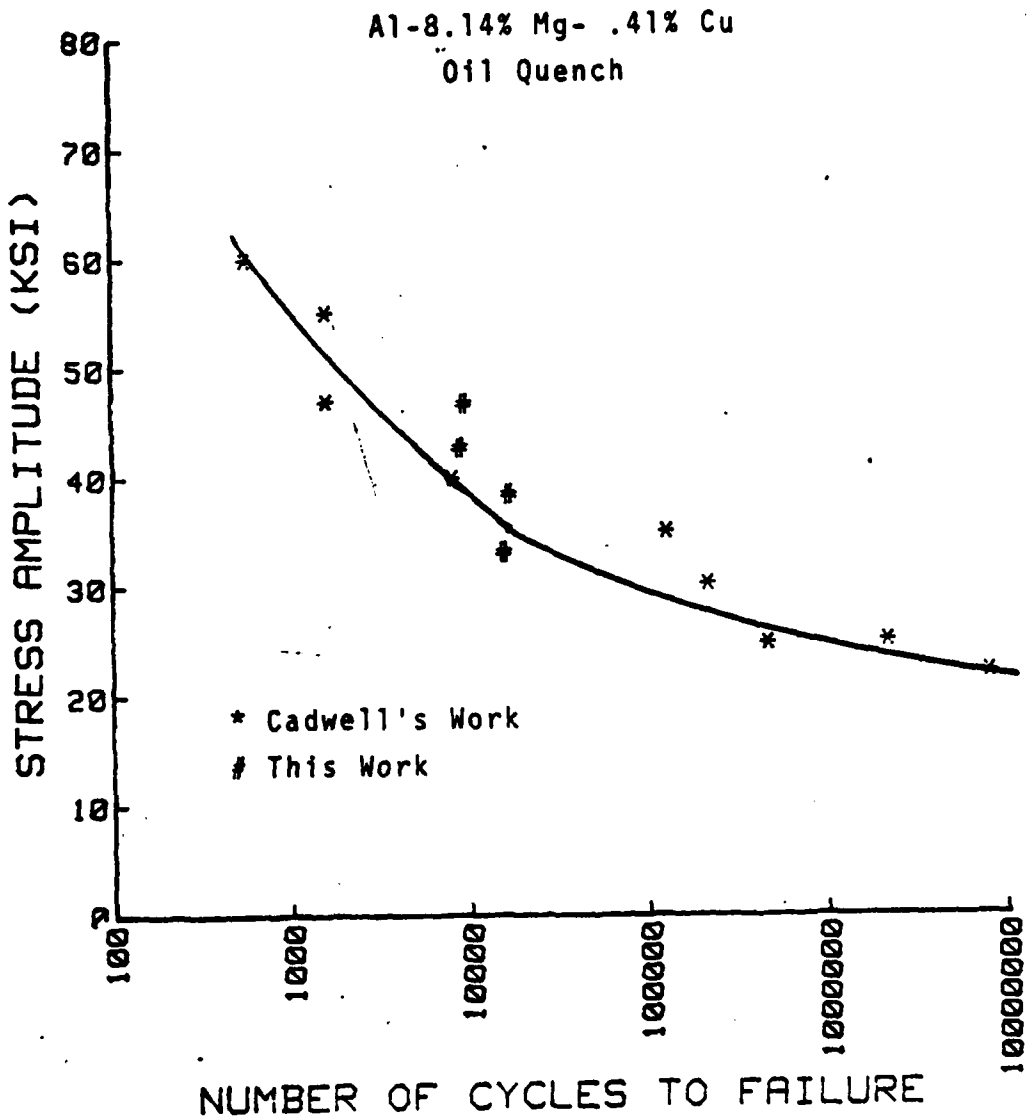


Figure 16. Stress-Amplitude vs Cycles-to-Failure for Al-8.14%Mg-0.41%Cu, solution treated and forged @ 440°C, oil quenched and warm rolled @ 300°C; *Cadwell's work [Ref. 8] - 10-hour solution treatment, #This work - 24-hour solution treatment.

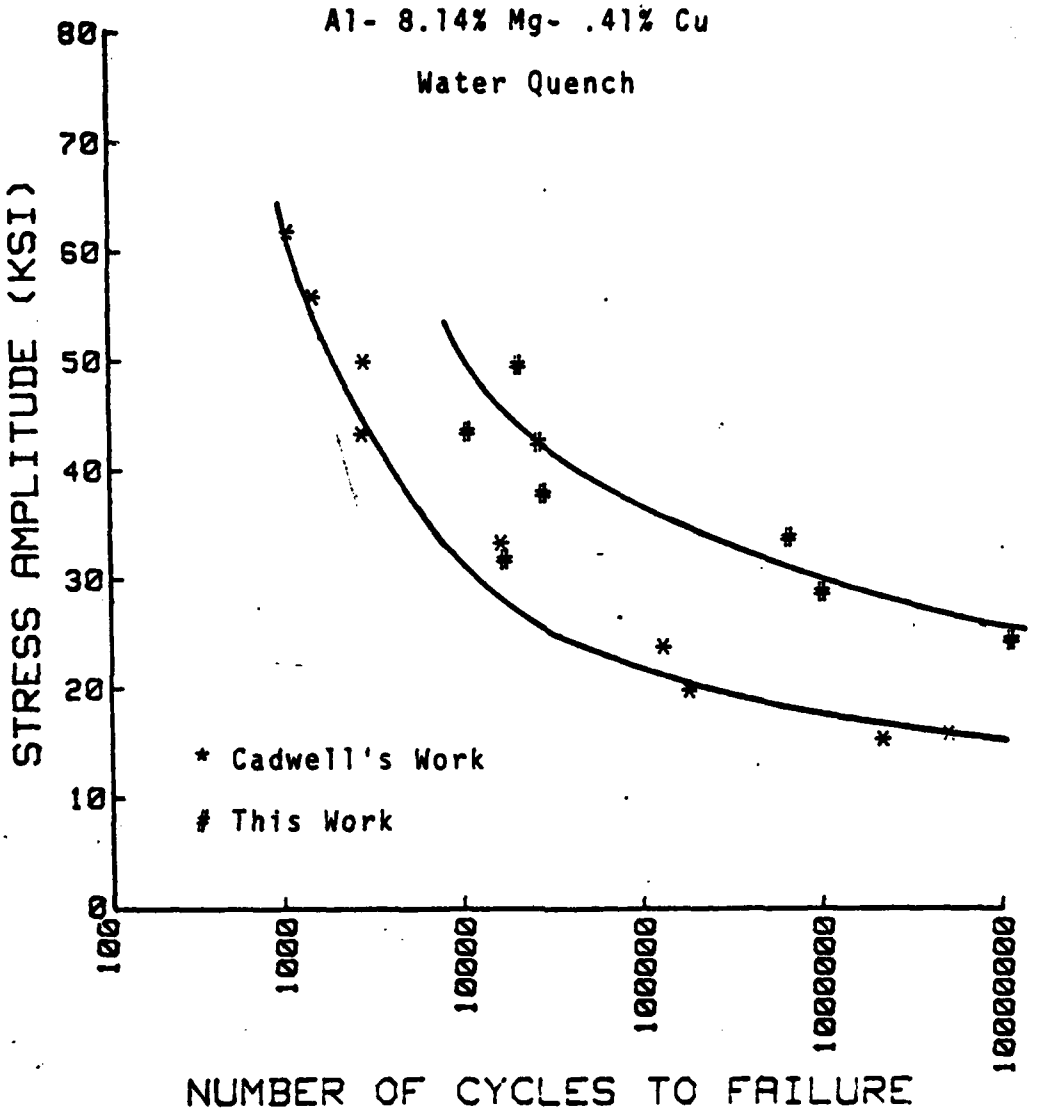


Figure 17. Stress-Amplitude vs Cycles-to-Failure for Al-8.14%Mg-0.41%Cu, solution treated and forged @ 440°C, water quenched and warm rolled @ 300°C; *Cadwell's work [Ref. 8] - 10-hour solution treatment, #This work - 24-hour solution treatment.

respectively. The data of this work coincides closely with that of Cadwell [Ref. 8] suggesting no effect attributable to increasing the solution treatment time for the oil quenched condition. In contrast, increased solution treatment time has a dramatic effect on the fatigue response for the water quenched condition. The fatigue life at any stress is greater with the longer solution treatment time and under high cycle fatigue (HCF) conditions, the stress to obtain a life of 10^7 cycles is increased by nearly 50%, from 117 Mpa (17 Ksi) to 172 Mpa (25 Ksi).

Figures 18 and 19 present results for the Al-10.2%Mg alloy in the oil and water quenched conditions, respectively. The data obtained in this work now coincides with that of Cadwell [Ref. 8] for both conditions, i.e. for both oil and water quenched conditions. The increased solution treatment time has had no apparent effect as opposed to the large effect noted for the Al-8.14%Mg-0.41%Cu alloy in the water quenched conditions.

Cadwell [Ref. 8] attributed the generally good fatigue resistance of these alloys to the presence of a dispersion of micron-sized intermetallic β particles in the alloy. These particles were envisioned to disperse slip and make more difficult the formation of persistent slip bands in the manner described by Martin and his co-workers [Refs. 16,17]. Better fatigue resistance in the oil quenched condition of both alloys was attributed to the formation of more coarse particles in the slower quench when compared to the water quenched condition.

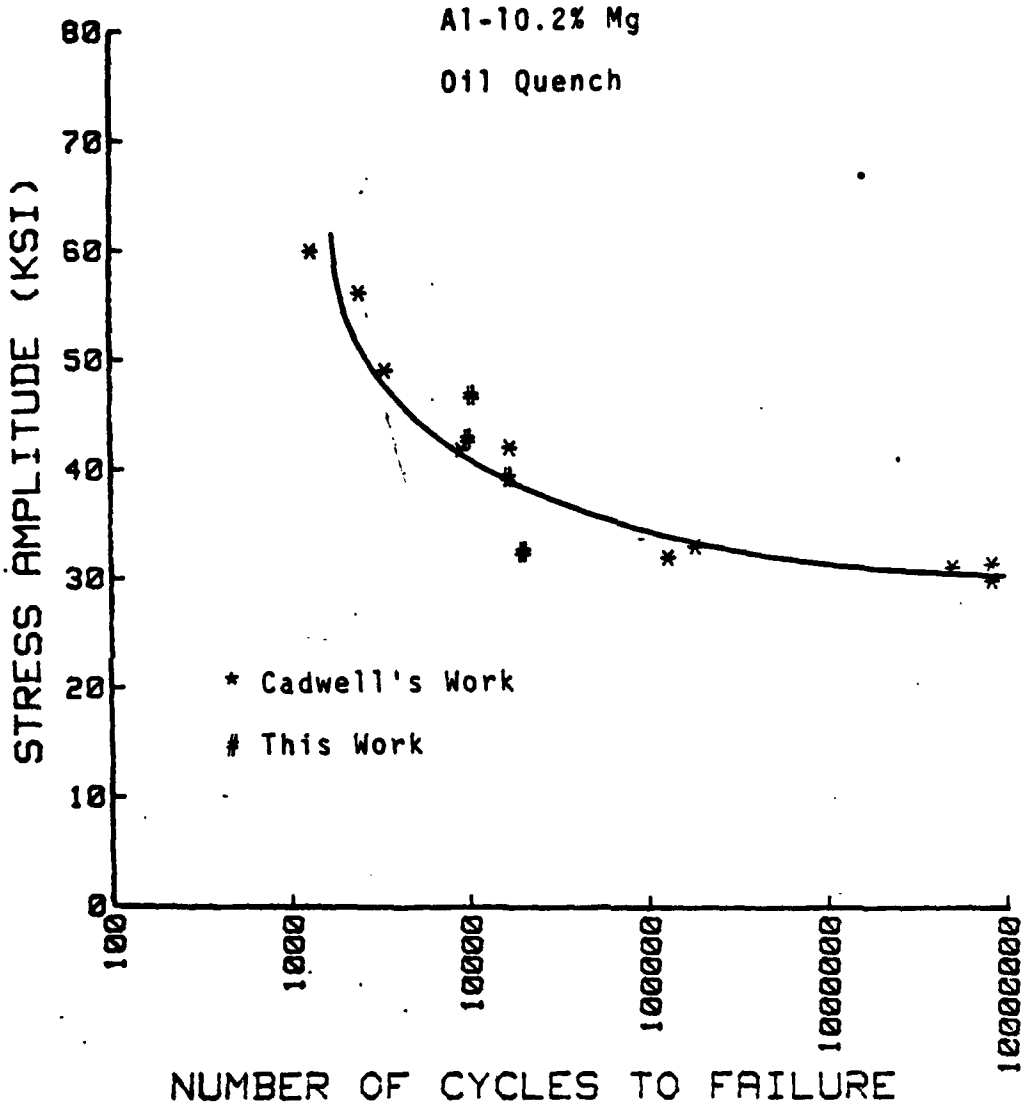


Figure 18. Stress-Amplitude vs Cycles-to-Failure for Al-10.2%Mg, solution treated and forged @ 440°C, oil quenched and warm rolled; *Cadwell's work [Ref. 8] - 10-hour solution treatment, #This work - 24-hour solution treatment.

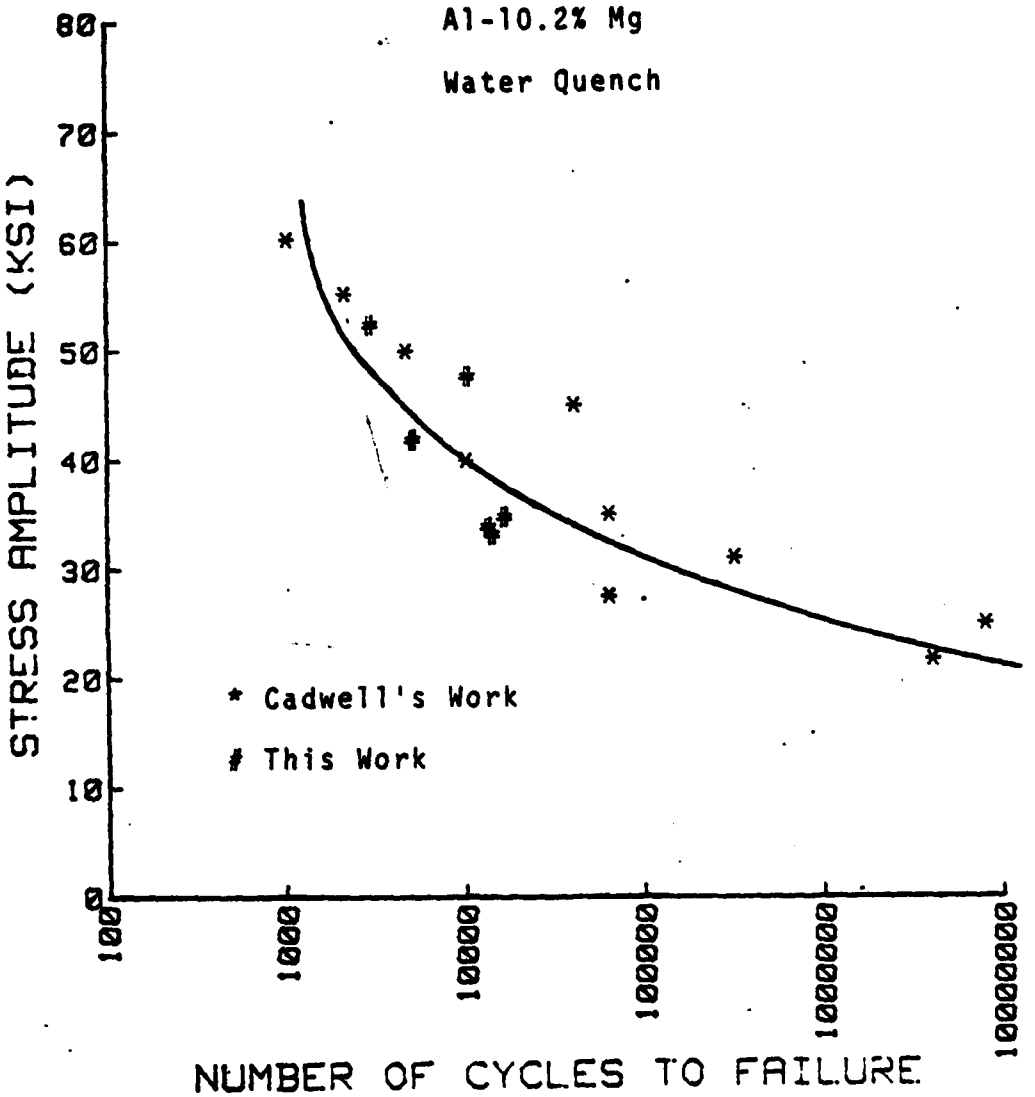


Figure 19. Stress-Amplitude vs Cycles-to-Failure for Al-10.2%Mg, solution treated and forged @ 440°C, water quenched and warm rolled; *Cadwell's work [Ref. 8] - 10-hour solution treatment, #This work - 24-hour solution treatment.

The microstructural examination conducted in this work has demonstrated an additional, important feature of this processing, namely that grain boundary precipitation accounts for much of the precipitated Mg, both for short time (10 hours) solution treatments and long time (24 hours) solution treatment followed by oil quenching. In fact, grain boundary precipitation is still evident even in the 24-hour solution treatment and water quenched condition of the Al-10.2%Mg alloy. Only the Al-8.14%Mg-0.41%Cu alloy exhibits little or no tendency to this phenomenon when given this 24-hour solution treatment with water quenching. This may be verified by comparing the optical micrographs obtained in this work to those presented by Cadwell [Ref. 8] in his work on these same two alloys. This observation suggests that the grain boundary precipitates may be more effective in prevention of slip band formation when such precipitates are somewhat coarser (oil quenched) than when finer (water quenched). However, the effectiveness of the intermetallic when uniformly dispersed was not evaluated in Cadwell's work [Ref. 8]. In this effort, the extension of solution treatment time to 24 hours, followed by water quenching, did result in a more nearly uniform precipitate dispersion with little intergranular precipitation but only for the Al-8.14%Mg-0.41%Cu alloy. The fatigue data obtained (Fig. 17) demonstrates the importance of this microstructural homogenization and suggests that the intermetallic precipitate is still more effective when uniformly dispersed than when present

intergranularly. The remaining conditions represented in Figures 16, 18 and 19 are all microstructures influenced by intergranular precipitation. These intergranular precipitates are effective in promoting fatigue resistance as suggested by the data for the Al-10.2%Mg alloy, especially as oil quenched. Nonetheless, the data for the Al-8.14%Mg-0.41%Cu alloy suggest the intermetallic is still more effective when uniformly dispersed. This result is also consistent when comparison is made between the results of Cadwell [Ref. 8] and of this work in the area of tensile mechanical properties. Previously, it was reported [Ref. 8] that the ductility of the Al-8.14%Mg -0.41%Cu alloy was sensitive to quench rate when the solution treatment time was ten hours. Here, the two quench rates resulted in essentially the same ductility for both quench rates but longer solution treatment. This, too, is considered to result from the improved homogeneity attained in the longer solution treatment of the material.

Finally, the Al-10.2%Mg-0.52%Mn alloy was not processed in sufficient quantity to produce significant fatigue data. This alloy, however, possesses a particularly homogeneous microstructure and, based on the foregoing discussion, may be expected to have excellent fatigue resistance. This area should receive more attention in future work on this alloy system.

V. CONCLUSIONS AND RECOMMENDATIONS

The objective of this research was to investigate the influence of solution treatment time and quench rate on microstructure, strength and fatigue resistance of warm-rolled Aluminum-Magnesium alloys. This objective was attained and the following conclusions are drawn:

1) Increased solution treatment time results in improvement of microstructural homogeneity, especially in the Cu-containing and Mn-containing alloys, but that quench rate is also critically important;

2) High quench rates are necessary (water as opposed to oil) to retain Mg in solution prior to warm-rolling; alloy content is important in that Cu in an 8%Mg alloy and Mn in a 10%Mg alloy appear to assist in retention of Mg in solution and thereby allow for more homogeneous precipitation;

3) Solution treatment time and quench rate have minor influence on tensile mechanical properties;

4) Solution treatment time and quench rate have a significant effect on the resulting fatigue behavior when the Mg is held in solution prior to warm-rolling and then precipitated uniformly during warm-rolling.

5) The addition of Mn to a 10%Mg alloy results in refinement and homogenization and development of a fine substructure

in warm-rolling. Resulting mechanical properties were a yield strength of 393 Mpa (57 Ksi), ultimate tensile strength of 517 Mpa (75 Ksi) with 11% elongation to fracture.

Recommendations for further work are:

1) Additional Transmission Electron Microscopy study of microstructural development in these alloys with particular attention to the Mn-containing materials.

2) Characterization of fatigue in the Mn-containing alloys in conjunction with more detailed study of solution treatment time, temperature and quench rate effects.

3) Investigation of solution treatment cycles involving higher temperatures than the 440^{o}C currently employed.

TABLE I.

Table of Thermo-mechanical Processes Mechanical Testing Results from Ref. 7

Alloy 1 Al-8.14%Mg-0.41%Cu

Alloy 2 Al-10.2%Mg

Alloy 3 Al-10.2%Mg-0.52%Mn

These alloys solution treated for 10 hours @ 440°C, forged, oil quenched, and warm-rolled at 300°C.

Alloy	Ultimate Tensile Strength		0.2% Offset Yield Strength		Elongation	Hardness
	Mpa	KSI	Mpa	KSI	%	R_B
1	479	69.5	383	55.5	10.3	73.0
2	472	68.5	371	53.9	10.8	76.0
3	580	84.2	470	68.2	7.3	91.0

TABLE II.

Table of Thermo-mechanical Processes Mechanical Testing Results from Ref. 8

Alloy 1 Al-8.14%Mg-0.41%Cu

Alloy 2 Al-10.2%Mg

These alloys solution treated for 10 hours @ 440°C, forged, quenched and warm rolled at 300°C.

Alloy	Quench	Ultimate Tensile Strength		0.2% Offset Yield Strength		Elongation
		Mpa	KSI	Mpa	KSI	%
1	Oil	448	65	353	51	10.5
1	Water	448	65	353	51	14.8
2	Oil	445	64.6	317	46	16.2
2	Water	487	70.6	317	46	16.5

TABLE III.

Table of Thermo-mechanical Processes Mechanical Testing Results from this Work

Alloy 1 Al-8.14%Mg-0.41%Cu

Alloy 2 Al-10.2%Mg

Alloy 3 Al-10.2%Mg-0.52%Mn

These alloys solution treated for 10 or 24 hours @ 440°C, forged, oil or water quenched and warm-rolled at 300°C.

Solution Treatment Time HRS	Quench	Alloy	Ultimate Tensile Strength		0.2% Offset Yield Strength		Elongation	Hardness
			Mpa	KSI	Mpa	KSI	%	R_B
10	Oil	1	446	64.8	352	51.2	16.3	71.7
24	Oil	1	432	62.8	320	46.5	18.0	71.5
24	Water	1	436	63.4	336	48.9	19.0	70.3
10	Oil	2	449	65.3	336	48.9	20.0	71.2
24	Oil	2	438	63.7	325	47.3	19.0	68.6
24	Water	2	455	66.1	350	50.9	17.0	71.0
24	Water	3	522	75.8	398	57.8	11.0	79.0

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