THERMOMECHANICAL PROCESSING AND AMBIENT TEMPERATURE PROPERTIES OF A 6061 ALUMINUM 10 VOLUME PERCENT ALUMINA METAL MATRIX COMPOSITE

by

Thomas A. Schaefer

March 1990

Thesis Advisor

T.R. Mc Nelley

Approved for public release; distribution is unlimited.
Thermomechanical processing was conducted on a cast aluminum-based metal matrix composite. The material studied was a 6061 aluminum containing 10 volume percent of alumina (Al₂O₃) particles, fabricated by casting and subsequently extruded by DURALCAN, Inc. Processing included isothermal rolling of an extruded bar to large strain values. As a result of rolling at 500°C, strength was increased, but with a substantial loss of ductility. Further strengthening was realized by rolling at 350°C and no further ductility loss was seen. Homogeneity of the particle dispersion was considerably improved with no evidence of microstructural damage. Upon subsequent solution heat treatment, ductility of the rolled materials was restored to values greater than obtained in material experiencing only extrusion. Also, the strength of the rolled material exceeded that of material heat treated after extrusion. Upon subsequent aging treatment (aging at 160°C), the increased strength and ductility enhancement persisted.
Thermomechanical Processing and Ambient Temperature Properties of a 6061 Aluminum 10 Volume Percent Alumina Metal Matrix Composite

by

Thomas A. Schaefer
Lieutenant, United States Navy
B.S., United States Naval Academy, 1984

Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN MECHANICAL ENGINEERING

from the

NAVAL POSTGRADUATE SCHOOL
March 1990

Author: Thomas A. Schaefer

Approved by: T.R. Mc Nelley, Thesis Advisor

Anthony J. Healey, Chairman,
Department of Mechanical Engineering
ABSTRACT

Thermomechanical processing was conducted on a cast aluminum-based metal matrix composite. The material studied was a 6061 aluminum containing 10 volume percent of alumina (Al₂O₃) particles, fabricated by casting and subsequently extruded by DURALCAN, Inc. Processing included isothermal rolling of an extruded bar to large strain values. As a result of rolling at 500°C, strength was increased, but with a substantial loss of ductility. Further strengthening was realized by rolling at 350°C and no further ductility loss was seen. Homogeneity of the particle dispersion was considerably improved with no evidence of microstructural damage. Upon subsequent solution heat treatment, ductility of the rolled materials was restored to values greater than obtained in material experiencing only extrusion. Also, the strength of the rolled material exceeded that of material heat treated after extrusion. Upon subsequent aging treatment (aging at 160°C), the increased strength and ductility enhancement persisted.
TABLE OF CONTENTS

I. INTRODUCTION .................................................. 1

II. BACKGROUND .................................................... 3
   A. REINFORCEMENT USED IN DISCONTINUOUS METAL MATRIX COMPOSITES ........... 3
   B. MATRIX ALLOY ....................................... 4
   C. DISCONTINUOUS METAL MATRIX COMPOSITES ........................................ 5
   D. POST FABRICATION PROCESSING OF METAL MATRIX COMPOSITES .................... 6
   E. HEAT TREATMENT OF DISCONTINUOUS METAL MATRIX COMPOSITES .................... 7

III. EXPERIMENTAL PROCEDURE .................................... 8
   A. MATERIAL AND SECTIONING ..................................... 8
   B. THERMOMECHANICAL PROCESSING ................................... 8
   C. MACHINING .................................................................. 9
   D. TENSILE TESTING .................................................. 9
   E. DATA REDUCTION .................................................. 9
   F. OPTICAL MICROSCOPY ............................................ 10
   G. AGE HARDENING STUDY ......................................... 11

IV. RESULTS AND DISCUSSION ...................................... 13
   A. AS CAST CONDITION ............................................. 13
   B. PROCESSED CONDITIONS .......................................... 15
      1. Microstructural Effects of Processing ........................................... 15
      2. Effects of Processing upon Mechanical Properties ................................... 15
      3. Effects of Processing on Particle Size ............................................. 16
   C. AGE HARDENING STUDY .......................................... 25
      1. Solution Treatment ............................................... 25
      2. The Results of Aging at 160°C ................................................. 25
   D. SUMMARY .......................................................... 25
V. CONCLUSIONS .......................................................... 32
VI. RECOMMENDATIONS FOR FURTHER STUDY .............. 33
APPENDIX STRESS STRAIN CURVES .............................. 34
LIST OF REFERENCES ..................................................... 44
INITIAL DISTRIBUTION LIST ........................................... 46
LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PROPERTIES OF COMMONLY USED DISCONTINUOUS REINFORCEMENT</td>
<td>3</td>
</tr>
<tr>
<td>2</td>
<td>COMPOSITION LIMITS FOR 6061 ALUMINUM IN WEIGHT PERCENT</td>
<td>4</td>
</tr>
<tr>
<td>3</td>
<td>COMPARISON OF REINFORCED AND UNREINFORCED 6061 ALUMINUM</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>ROLLING SCHEDULE</td>
<td>12</td>
</tr>
<tr>
<td>5</td>
<td>SAMPLE PREPARATION</td>
<td>12</td>
</tr>
<tr>
<td>6</td>
<td>MECHANICAL PROPERTIES FOLLOWING AGING TO PEAK STRENGTH</td>
<td>27</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Figure 1. Endurance Limit Comparison ........................................... 5
Figure 2. Tensile Test Specimen Drawing ......................................... 10
Figure 3. Specimen Planes of View .................................................. 11
Figure 4. As Cast Condition .............................................................. 13
Figure 5. Stress-Strain Curves for Cast Condition ................................. 14
Figure 6. Triplanar Optical Micrograph of Extruded Condition .................. 17
Figure 7. Triplanar Optical Micrograph of 500°C Rolled Condition ............... 18
Figure 8. Triplanar Optical Micrograph of 350°C Rolled Condition .............. 19
Figure 9. Stress-Strain Curves of the Processed Conditions ...................... 20
Figure 10. As Cast Condition ............................................................ 21
Figure 11. Extruded Condition .......................................................... 22
Figure 12. 500°C Rolled Condition ..................................................... 23
Figure 13. 350°C Rolled Condition ..................................................... 24
Figure 14. Stress-Strain Comparison After Solution Treatment ................. 28
Figure 15. 1% Offset Yield Strength vs. Aging Time ............................. 29
Figure 16. Ultimate Tensile Strength vs. Aging Time ............................ 30
Figure 17. Percent Elongation vs. Aging Time .................................... 31
Figure 18. Stress-Strain Curves for Extruded Material ........................... 34
Figure 19. Stress-Strain Curves for 500°C Rolled Material ....................... 35
Figure 20. Stress-Strain Curves for 350°C Rolled Material ....................... 36
Figure 21. Stress-Strain Curves for 10 minute (T6) Heat Treatment ............. 37
Figure 22. Stress-Strain Curves for 30 minute (T6) Heat Treatment ............. 38
Figure 23. Stress-Strain Curves for 1 hour (T6) Heat Treatment ............... 39
Figure 24. Stress-Strain Curves for 4 hour (T6) Heat Treatment ............... 40
Figure 25. Stress-Strain Curves for 8 hour (T6) Heat Treatment ............... 41
Figure 26. Stress-Strain Curves for 16 hour (T6) Heat Treatment .............. 42
Figure 27. Stress-Strain Curves for 120 hour (T6) Heat Treatment ............... 43
I. INTRODUCTION

A renewed interest in the development of discontinuous metal matrix composites (MMCs) has emerged due to recent achievements in fabrication methods including development of lower cost techniques. MMCs constitute a family of materials consisting of hard, brittle fine particles or short fibers embedded in a soft more ductile matrix [Ref. 1]. The reinforcing particles or fibers act to refine the structure and act as obstacles to dislocation movement. The result is a stronger material than the unreinforced counterpart. Also, the modulus of elasticity is increased resulting in a stiffer material as well.

Specific properties of MMCs can be tailor made to satisfy the requirement of a wide array of design applications. This is accomplished through careful selection of the base matrix, the size, and the quantity of reinforcement [Ref. 2]. Some of the material properties that can be controlled in this manner are listed below:

- modulus
- density
- elevated temperature strength
- thermal and electrical conductivities
- corrosion and abrasion resistance.

Furthermore, mechanical anisotropy often present in continuous fiber reinforced composites is not present in a well fabricated discontinuous metal matrix composite. Adding to this impressive list, lower fabrication costs, have made MMCs an ideal choice in applications where unreinforced materials come up short on properties.

The fabrication of discontinuously reinforced MMCs traditionally had been accomplished through powder metallurgy (P.M) techniques. Powder metallurgy involves the blending of fine powders of both the base matrix and reinforcement. The two are then consolidated under high pressure and ultimately sintered at elevated temperature. Due to the fact that the matrix powder is usually larger than the particulate reinforcement the material must be further worked to ensure a uniform reinforcement distribution. Thus P.M techniques are rather expensive, time consuming, and product size is limited. Recently ingot metallurgy techniques (casting) have become more widely recognized as a fabrication method. The casting of MMCs involves direct mixing of the reinforcement with the molten matrix. This technique is potentially much easier, faster and, not least,
cheaper. Product size is not limited and the method is suitable for a variety of base matricies and reinforcements [Ref. 3]. Although the casting of MMCs offer these advantages, it also has the following potential problems:

- poor wetting of reinforcement surface and melt
- chemical reaction of reinforcement with melt
- non-uniform reinforcement distribution [Refs. 4,5].

The combination of all these problems may lead to poor material properties.

Dural Aluminum Composites Corporation (DURALCAN) located in San Diego, California, is a leader in the production of discontinuous cast MMCs. DURALCAN has developed a proprietary casting process which has addressed the common problem areas. The process involves a pretreatment which allows chemically active molten aluminum to wet the particles without reacting with and thereby degrading the particles. In addition, the pretreatment lessens dissolved gases and aids in the formation of a more homogenous microstructure. The end result is a good quality composite comparable to one produced by powder metallurgy [Ref. 6].

The production of a good quality cast MMC has opened the door for further research on these unique materials. Of particular interest here are the effects of thermomechanical processing upon mechanical properties and microstructural characteristics. It was the purpose of this thesis to conduct an initial investigation of the effects of thermomechanical processing upon a DURALCAN supplied metal matrix composite.
II. BACKGROUND

A. REINFORCEMENT USED IN DISCONTINUOUS METAL MATRIX COMPOSITES

The purpose of the reinforcement is to increase strength and stiffness relative to the metallic matrix. Forms commonly used are short, chopped fibers (whiskers) or fine particles. These fibers or particles alone have no structural value by themselves. They are extremely high in strength and their stiffness is significantly greater than the base matrix alloy. However, the fibers or particles must be consolidated with a matrix to transmit structural loads. The increase in stiffness is perhaps the most important result of added reinforcement. In fact in a discontinuous aluminum metal matrix composite, with only modest reinforcement content (15%), has a modulus equivalent to that of a relatively costly titanium aircraft alloy [Ref. 7].

The fibers and particles used as reinforcement are abundantly available since most are derivatives of common refractory fibers. The costs of discontinuous fibers and particles are relatively low when compared to those associated with continuous fiber reinforcement. For example, discontinuous alumina fiber ranges in price from 35-90 $ \text{kg}^{-1}$, whereas continuous fiber reinforcement on average costs between 165 and 330 $\text{kg}^{-1}$ [Ref. 8].

The reinforcement used in the MMC supplied by DURALCAN was a fine alumina particulate approximately 10 micron in diameter. Properties of common discontinuous reinforcement are shown in Table 1 below [Ref. 9].

<table>
<thead>
<tr>
<th>Material</th>
<th>Young's Modulus (GPa)</th>
<th>Density (g cm$^{-3}$)</th>
<th>Thermal Conductivity (W m$^{-1}$K)</th>
<th>Coeff. of Thermal Expansion ($10^{-6}$°K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon Carbide</td>
<td>400</td>
<td>3.21</td>
<td>120</td>
<td>3.4</td>
</tr>
<tr>
<td>Alumina</td>
<td>379</td>
<td>3.98</td>
<td>30</td>
<td>7.0</td>
</tr>
<tr>
<td>Aluminum Nitride</td>
<td>345</td>
<td>3.26</td>
<td>150</td>
<td>3.3</td>
</tr>
</tbody>
</table>
B. MATRIX ALLOY

The role of the matrix in a metal matrix composite is to serve as the glue to bind together the material. The matrix must bond with the reinforcement and must distribute the load evenly. The most widely used matrices for MMCs are medium- and high-strength aluminum based alloys. Some examples of those used are: 2014, 2024, 4032, 6061, and 7075 aluminum alloys. Careful attention must be taken when selecting a matrix, particularly in cast MMCs. One must ensure that any adverse chemical reactions do not take place between the matrix and the chosen reinforcement. If such reactions do take place they may seriously degrade the reinforcement and ultimately the material quality [Ref. 10].

The 6061 aluminum alloy was the matrix of the MMC used in this research. This is a medium strength, heat treatable aluminum alloy. The main alloying constituents are magnesium and silicon, which combine to form a magnesium silicide precipitate and thus make the alloy heat treatable. The 6061 alloy is commonly heat treated to one of two different heat treatments. The T4 treatment is a natural aging process where the alloy attains peak strength within a few days at ambient temperature. The T6 treatment is an artificial aging scheme conducted at 160°C. The alloy is held at this temperature for a specified time period to improve both strength and hardness. The aging time to peak strength for the 6061-T6 is 18 hours [Ref. 11]. The nominal composition limits are listed in Table 2 below [Ref. 12].

Table 2. COMPOSITION LIMITS FOR 6061 ALUMINUM IN WEIGHT PERCENT

<table>
<thead>
<tr>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Zn</th>
<th>Ti</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>to</td>
<td>0.7</td>
<td>to</td>
<td>0.15</td>
<td>to</td>
<td>0.8</td>
<td>to</td>
<td>0.04</td>
</tr>
<tr>
<td>0.8</td>
<td>max</td>
<td>0.40</td>
<td>max</td>
<td>0.15</td>
<td>max</td>
<td>0.12</td>
<td>max</td>
<td>0.25</td>
</tr>
<tr>
<td></td>
<td>max</td>
<td></td>
<td></td>
<td></td>
<td>max</td>
<td></td>
<td></td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.05</td>
</tr>
</tbody>
</table>

6061 is a versatile aluminum alloy. It possesses good formability and is used in applications where moderate strength combined with weldability and corrosion resistance are necessary. Examples of its uses are the following:

- trucks
- marine applications
railroad cars
• pipelines [Ref. 13].

Due to the advantages discussed above, 6061 has become a popular choice as a base matrix alloy of MMCs for both P/M and cast fabrication methods.

C. DISCONTINUOUS METAL MATRIX COMPOSITES

The addition of reinforcement to the metallic matrix results in a significantly stronger and stiffer material. The increase in stiffness is seen as the greatest plus for MMCs, since this is a property which cannot be improved through alloying. DURALCAN, has taken full advantage of this property. They have recently produced material for a tennis racket that provides 25% reduced vibration and increased stiffness at a lower cost compared with graphite-epoxy rackets [Ref. 2]. In addition, due to the high strength-to-weight ratio, MMCs are currently being evaluated for use in automobile and aerospace applications [Ref. 8]. Questions still remain concerning the fatigue and fracture characteristics of MMCs. Fatigue and fracture characteristics must be competitive with those of unreinforced alloys before MMCs can be seriously considered for many aerospace and automobile applications. The associated problems and higher costs in fabrication and subsequent processing are the primary reasons for this. Data in these areas is scarce at best. However, from what has been reported, it appears that the endurance limit is more highly dependent upon the matrix alloy used rather than the amount or type of reinforcement [Ref. 8]. Figure 1 illustrates a comparison of endurance limits of unreinforced aluminum alloys to those of discontinuous aluminum silicon carbide MMCs [Ref. 8].

![Figure 1. Endurance Limit Comparison](attachment:image.png)
The discontinuous MMC of primary concern in this research was cast and extruded 6061 aluminum containing 10 volume percent alumina ($Al_2O_3$). Table 3 compares the mechanical properties of 6061-T6 and the supplied 6061 aluminum 10 volume percent alumina MMC [Ref. 14].

Table 3. COMPARISON OF REINFORCED AND UNREINFORCED 6061 ALUMINUM

<table>
<thead>
<tr>
<th>Material</th>
<th>UTS (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation (%)</th>
<th>Elastic Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061-T6</td>
<td>310(max) 262(min)</td>
<td>276(max) 241(min)</td>
<td>20</td>
<td>68.9</td>
</tr>
<tr>
<td>10(^{\circ}%)\ Al_2O_3</td>
<td>338(max) 255(min)</td>
<td>296(max) 255(min)</td>
<td>7.6</td>
<td>81.4</td>
</tr>
</tbody>
</table>

D. POST FABRICATION PROCESSING OF METAL MATRIX COMPOSITES

The primary purpose of post solidification processing is to improve the homogeneity of the reinforcement distribution. In doing so it is anticipated that both strength and ductility will be improved. Normally, this is accomplished via extrusion, usually with an extrusion ratio of at least 10:1 and more commonly 20:1 [Ref. 10]. Following extrusion, the material can then be rolled or forged to improve microstructural homogeneity to an even greater degree.

Some work has been reported on the effects of thermomechanical processing upon discontinuous MMCs fabricated by P M techniques [Refs. 15,16,17]. Harrigan, et al.[Ref. 15], hot rolled a 6061 aluminum reinforced with silicon carbide particles to reductions of at least 80\(^{\circ}\). Dramatic improvements in both strength and ductility were noted. Maclean, et al. [Ref. 16], performed both rolling and extrusion on a 6061 aluminum reinforced with silicon carbide whiskers. Only slight improvement in strength and ductility were observed. Damage to reinforcement from processing is an important area of concern [Ref. 18]. Pickens, et al. [Ref. 17], processed both 7090 and 6061 aluminum reinforced with silicon carbide whiskers. It was noted in both materials that a large degree of reinforcement damage had taken place.

Processing studies of discontinuous cast MMCs are rare and little work has been reported in the literature. Dutta, et al. [Ref. 19], forged and hot rolled a cast 5083 alu-
minimum reinforced with silicon carbide particles. A considerable improvement in microstructure homogeneity and elongation to fracture were observed. However, no concurrent improvement in strength of the processed material was noted.

E. HEAT TREATMENT OF DISCONTINUOUS METAL MATRIX COMPOSITES

Following thermomechanical processing, heat treatment can be used to attain optimum material mechanical properties. The heat treatments conducted are of the same type as those used for unreinforced matrix alloy. Results from research on heat treatment response have shown that discontinuous MMCs age at a much faster rate than their unreinforced counterparts [Ref. 10].
III. EXPERIMENTAL PROCEDURE

A. MATERIAL AND SECTIONING

The 6061 Aluminum with 10 volume percentage alumina (Al₂O₃) MMC was provided in both the cast and the as-extruded conditions by Dural Aluminum Composites Corporation (DURALCAN). The cast material was in the form of a plate with dimensions 102 mm x 98 mm x 13 mm (4.1 in x 3.85 in x 0.5 in). A single portion of dimensions 51mm x 47mm x 13mm (2.0 in x 1.85 in x 0.5 in) was sectioned from the plate for subsequent machining of tensile specimens.

The extruded material was obtained from the cast material which was subjected to a 17:1 extrusion [Ref. 20]. This corresponds to a strain $\varepsilon = 2.83$. It was in the form of a rectangular bar with rounded edges and dimensions 76 mm x 19 mm (3.0 in x 0.75 in). Three sections of the bar with dimensions 76 mm x 47 mm x 13 mm (3.0 in x 2.5 in x 0.75 in) were removed for subsequent processing and machining of tensile specimens. Two of these were further sectioned to dimensions 38 mm x 64 mm x 19 mm (1.5 in x 2.5 in x 0.75 in) for processing as rolling billets. A power hack saw was used for all material sectioning. Scrap material was cut into small coupons for use in optical microscopy.

B. THERMOMECHANICAL PROCESSING

Solution treatment for 90 minutes at 560°C to ensure microstructural homogenization of the matrix was accomplished using a Lindberg type B-6 Heavy Duty Furnace. All billets were immediately quenched to room temperature in water following solution treatment.

Thermomechanical processing (TMP) in the form of isothermal rolling was conducted at two different temperatures, 500°C and 350°C, to further strain $\varepsilon = 2.24$. The use of two different rolling temperatures served as a means to vary dislocation density imparted to each billet.

Each billet was placed in a Blue M furnace, model 8655F-3, to provide heating at the rolling temperature for 30 minutes prior to the first rolling pass. This allowed the billet to equilibrate at the desired rolling temperature.

All billets were rolled utilizing a Fenn Laboratory Rolling mill. The rolling schedule used is summarized in Table 4. The schedule was similar to those developed for processing of superplastic Al-Mg alloys [Ref. 21]. It was found after two unsuccessful at-
tempts at rolling that a spray silicone lubricant was necessary to ensure trouble free rolling. No lubricant was used during the first attempt to roll the MMC. On the fifth pass the billet stuck to the rolls and much time and effort was required to remove it. In an effort to avoid this result, a spray graphite lubricant was used for the second attempt. This had the opposite effect. The graphite lubricant reduced the friction to such a degree that the billet could not be fed into the mill at all. The spray silicone lubricant proved to be an excellent compromise. It eliminated the sticking problem, and it made cleaning of the rolls following each pass much easier. The silicone was applied prior to the fifth pass and every pass thereafter until completion of the rolling. Following each rolling pass the billets were returned to the furnace for 30 minutes of annealing. At the completion of the final rolling pass the billets were quenched in water to ambient temperature.

C. MACHINING

Billets of all three material conditions were machined to the dimensions for tensile testing as shown in Figure 2. Machining of the material proved to be rather difficult and resulted in excessive wear to machine tooling. The hard alumina particles within the matrix acted as an abrasive and quickly damaged standard carbide endmill cutting bits. The use of dulled tooling resulted in the presence of residual stresses in the finished tensile specimens. Many samples were actually warped during the machining process. DURALCAN recommends the use of diamond tipped tooling for ease of machining and extended tool life [Ref. 14]. Presently this type of tooling is on order for future research.

D. TENSILE TESTING

Tensile testing was performed on an Instron Model 6027 test machine. Output from the Instron test machine was linked to a Hewlett Packard Model 3852A Data Acquisition Unit, from which a graph of load vs. time was plotted. Tests were conducted at ambient temperature at a constant crosshead speed of 0.508 mm min.

E. DATA REDUCTION

Engineering stress vs. plastic strain plots were reduced from the generated graphs of load vs. time. In many tests grip slippage was observed. To compensate for this, the equation of the line which corresponded to the elastic region of the load vs. time curve was calculated. The distance from this line along with the corresponding load level was then converted to plastic strain and engineering stress.
F. OPTICAL MICROSCOPY

Optical microscopy was conducted on all material conditions to evaluate the alumina particle distribution. Samples were mounted in such a manner in order to facilitate study of three planes of view for each condition. These planes are defined as shown in Figure 3.

Conventional polishing techniques where in all scratches are removed before proceeding to the next step yielded poor results. In many instances, areas of relief were observed and in some cases the alumina particles were actually removed from the matrix. Better results were obtained by using timed intervals at each step and ensuring that only light pressure was applied during polishing. In addition it was found to be very helpful to ultrasonically clean the sample in ethyl alcohol and dry it after each polishing step. The procedural steps used in the sample preparation are listed in Table 5. A Zeiss ICM-405 Optical Microscope was used to take 35mm photographs.
G. AGE HARDENING STUDY

Since 6061 aluminum is an age hardenable alloy, a study was conducted to determine the effects of aging upon mechanical properties. A T-6 type aging scheme was used. Tensile specimens of both the extruded and rolled conditions were compared. All samples were first solution treated for 1 hour at 560°C. Following solution treatment, tensile specimens were quenched in water and then immediately placed in an aging furnace at 160°C. Specimens were removed at various time intervals, concluding with the overaged condition of 120 hours. Once removed all specimens were again quenched in water to room temperature and subjected to tensile tests at ambient temperature.
### Table 4. ROLLING SCHEDULE

<table>
<thead>
<tr>
<th>ROLL #</th>
<th>ROLL CHANGE (0.08in + 0.01in)</th>
<th>MILL SETTING (right left)</th>
<th>MILL GAP (in)</th>
<th>SILICONE LUBRICANT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>+ (8 + 0)</td>
<td>4.4</td>
<td>0.64</td>
<td>NO</td>
</tr>
<tr>
<td>2</td>
<td>-(1 + 2)</td>
<td>2.2</td>
<td>0.54</td>
<td>NO</td>
</tr>
<tr>
<td>3</td>
<td>-(1 + 2)</td>
<td>0.0</td>
<td>0.44</td>
<td>NO</td>
</tr>
<tr>
<td>4</td>
<td>-(1 + 2)</td>
<td>6.6</td>
<td>0.34</td>
<td>NO</td>
</tr>
<tr>
<td>5</td>
<td>-(1 + 2)</td>
<td>4.4</td>
<td>0.24</td>
<td>YES</td>
</tr>
<tr>
<td>6</td>
<td>-(0 + 6)</td>
<td>6.6</td>
<td>0.18</td>
<td>YES</td>
</tr>
<tr>
<td>7</td>
<td>-(0 + 6)</td>
<td>0.0</td>
<td>0.12</td>
<td>YES</td>
</tr>
<tr>
<td>8</td>
<td>-(0 + 5)</td>
<td>3.3</td>
<td>0.07</td>
<td>YES</td>
</tr>
<tr>
<td>9</td>
<td>-(0 + 2.2)</td>
<td>0.8 0.8</td>
<td>0.048</td>
<td>YES</td>
</tr>
</tbody>
</table>

### Table 5. SAMPLE PREPARATION

<table>
<thead>
<tr>
<th>STEP #</th>
<th>POLISHING MEDIUM</th>
<th>TIME</th>
<th>COMMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>320 GRIT</td>
<td>2 min</td>
<td>light pressure</td>
</tr>
<tr>
<td>2</td>
<td>400 GRIT</td>
<td>2 min</td>
<td>light pressure</td>
</tr>
<tr>
<td>3</td>
<td>600 GRIT</td>
<td>2 min</td>
<td>light pressure</td>
</tr>
<tr>
<td>4</td>
<td>6 micron diamond paste (metadi)</td>
<td>3 min</td>
<td>light pressure</td>
</tr>
<tr>
<td>5</td>
<td>3 micron diamond paste (metadi)</td>
<td>2 min</td>
<td>light pressure</td>
</tr>
<tr>
<td>6</td>
<td>colloidal silica</td>
<td>remove all scratches</td>
<td>light pressure wear gloves Highly caustic</td>
</tr>
</tbody>
</table>
IV. RESULTS AND DISCUSSION

A. AS CAST CONDITION

The as-cast condition's microstructure displayed a non-uniform reinforcement distribution. As seen in Figure 4, the alumina particles appear to be clustered and there are areas without reinforcement particles.

Figure 4. As Cast Condition: Relatively inhomogeneous distribution, with particle clustering and particle free areas. (200x)

Tensile test results were erratic and similar to those of an extremely brittle material. Percent elongation was on the order of 1% in most tests, and multiple fractures were commonplace. Figure 5 are representative stress-strain curves for the as-cast condition. These results likely reflect the non-uniform particle distribution.
Figure 5. Stress-Strain Curves for Cast Condition: Note the erratic behavior and extremely poor ductility. Maximum strain values on the order of 1-2%.
B. PROCESSED CONDITIONS

1. Microstructural Effects of Processing

The 17:1 extrusion \( (\varepsilon = 2.83) \) performed by DURALCAN has clearly homogenized the microstructure. Figure 6 shows there are still some areas of clustering, however overall a more uniform distribution of the \( \text{Al}_2\text{O}_3 \) particles is evident. Some banding of the particle distribution is apparent in both the long and short transverse planes of view.

Upon the introduction of additional strain \( (\varepsilon = 2.24) \), by isothermal rolling at 500\(^\circ\)C or 350\(^\circ\)C, the microstructural homogeneity was further improved. Figure 7 and Figure 8 clearly show this result. Banding in the direction of rolling is still evident in both the long and short transverse planes. Of noteworthy importance is the appearance that particle size has been reduced as well. Reasons for the appearance of smaller particles will be addressed later.

2. Effects of Processing upon Mechanical Properties

Tensile testing of the three processed conditions yielded some unexpected results. Figure 9 is a comparison of the displayed mechanical properties. Each stress-strain curve is a representative sample of a processed condition.

The extruded condition when compared to the as-cast form exhibited a marked increase in ductility. Strain to failure values in excess of 15\(^\%\) were obtained. However, ultimate tensile strength was reduced by approximately 18\(^\%\) when compared to the as-cast condition. The increase in ductility was attributed to the increased homogeneity of the microstructure.

The material rolled at 500\(^\circ\)C, when compared to the extruded condition, exhibited only a slight increase in strength, but a significant decrease in ductility. Ultimate tensile strength was enhanced by only 6\(^\%\), while ductility was degraded by almost one-half. The additional strain \( (\varepsilon = 2.24) \) imparted through rolling would lead one to expect a significant increase in strength, resulting from homogenization of particle distribution and refinement of the microstructure. A decrease in ductility was expected to accompany strengthening, but not the magnitude of decrease seen. The large decrease, however, was first thought to be related to damage to the microstructure caused by the rolling. Examination of the microstructure via optical microscopy did not support this conclusion. Upon consideration of the processing, rolling was performed below but near the solvus temperature for the 6061 matrix. This could have resulted in the formation of a coarse precipitate which would have adversely effected the mechanical properties. This requires further investigation.
The material rolled at 350°C displayed a significantly higher strength. However, ductility was comparable to that of material rolled at 500°C. In this condition strength was enhanced by 48% and ductility reduced by 37% compared to the extruded condition.

3. Effects of Processing on Particle Size

The processing has been shown to improve particle distribution in the matrix. However, comparison of the optical micrographs of the cast, extruded, and rolled conditions (Figure 4 and Figures 6-8) reveals that particle size appears to be reduced as strain increased. As greater strain was imparted to the material, the particles which make up clusters are spread further apart. The mean distance between particles is increased and therefore at lower magnifications, it appears that particle size is reduced. Figures 10-13 are higher magnification (500x) optical micrographs of the conditions in question. Viewing these in order of increased strain imparted, it appears that particle size has remained relatively constant throughout. In addition, and of equal importance, there seems to be no evidence that particles have been sheared or cracked due to processes imposed. Furthermore, there appears to be no evidence for decohesion at the particle matrix interfaces nor any sign of void formation within the matrix.
Figure 6. Triplanar Optical Micrograph of Extruded Condition: Extrusion has resulted in homogenization in all planes of view. Considerable banding of particles is evident in both the long and short transverse planes. (200x)
Figure 7. Triplanar Optical Micrograph of 500°C Rolled Condition: Rolling ($\epsilon = 2.24$) resulted in further homogenization of the particle distribution. Considerable banding is present in both the long and short transverse planes. (200x)
Figure 8. Triplanar Optical Micrograph of 350°C Rolled Condition: Rolling ($\epsilon = 2.24$) has also resulted in homogenization of the particle distribution. Considerable banding is still evident in both the long and short transverse planes. (200x)
Figure 9. Stress-Strain Curves of the Processed Conditions: Substantial improvement in ductility is observed in the extruded condition compared to the as-cast condition. Rolling at both 500°C and 350°C has increased strength, but with a substantial decrease in ductility.
Figure 10. As Cast Condition: Higher magnification optical micrograph (longitudinal view). Note clusters of particles with little or no separation between the individual particles of the cluster. (500X)
Figure 11. Extruded Condition: Higher magnification optical micrograph (longitudinal view). Note, as marked, the separation distance within clusters has increased due to extrusion. No evidence of particle shearing or breakage is apparent. (500X)
Figure 12. 500°C Rolled Condition: Higher magnification optical micrograph (longitudinal view). Note, as marked, separation distance has between particles has further increased due to rolling. Particle clusters appear to spread out within bands. No evidence of particle shearing or breakage is apparent as well. (500X)
Figure 13. 350°C Rolled Condition: Higher magnification optical micrograph (longitudinal view). Similar effects are noted here for material rolled at this lower temperature to those shown in Figure 12. (500X)
C. AGE HARDENING STUDY

1. Solution Treatment

Tensile tests conducted following one hour of solution treatment at 560°C yielded promising results. Upon subsequent solution treatment, the ductility of both rolled conditions was restored. In fact, the material rolled at 350°C is now more ductile than the extruded condition. Also, the strength of the rolled condition is slightly higher than that of the extruded material. These results may be attributed to microstructural homogenization due to rolling prior to the solution treatment. Figure 14 shows a comparison of the stress-strain curves for each of the processed conditions immediately following solution treatment.

2. The Results of Aging at 160°C

When comparing 1% offset yield strength throughout the subsequent aging interval some significant results clearly stand out. From Figure 15 it is evident that both of the rolled conditions exhibit a higher strength than the extruded condition throughout the entire aging interval. Also, both of the rolled conditions attain peak strength significantly faster than the extruded condition. Unreinforced 6061 aluminum aged at the same temperature (160°C) attains a peak yield strength of 276 MPa in 18 hours. Therefore, through the processes of extrusion and rolling the yield strength has been increased by approximately 40%. Increased ultimate tensile strength also persists, but the magnitude of difference was not as dramatic as that for yield strength (Figure 16).

Accompanying the increase in strength from rolling was an enhancement of ductility. Figure 17 shows that further processing has resulted in enhanced ductility over the entire aging period. With most processing schemes strength may be increased but ductility is then degraded. With this material, additional strain in processing followed by a subsequent heat treatment has resulted in both a stronger and more ductile final product.

D. SUMMARY

The as-cast material displayed mechanical properties characteristic of an extremely brittle material. This was attributed to the presence of a non-homogeneous particle distribution. Upon subsequent extrusion (ε = 2.83) homogeneity of the particle distribution was improved considerably. Associated with this, ductility of the extruded material was drastically improved. This is primarily attributed to the increased homogenization of the particle distribution. Additional factors include closure of
microporosity and elimination of coring, segregation and other related casting defects common to all solidified metallic alloys.

Isothermal rolling ($\varepsilon = 2.24$) conducted at $500^\circ C$ and $350^\circ C$ resulted in further homogenization of the particle distribution. Strength at ambient temperature for both rolling temperatures was improved. However, the ductility of the $500^\circ C$ rolled material was seriously degraded. This was believed to be a result of the proximity of the rolling temperature to the solvus temperature of the base matrix. It is proposed that rolling near the solvus temperature may result in the formation of a relatively coarse intermetallic precipitate of the $Mg_2Si$ phase, which would have deleterious effect upon mechanical properties. Another explanation was that rolling was having an adverse effect upon the alumina particles or the particle matrix interface and hence degrading the material properties. The optical microscopy showed no evidence of the latter and was limited in use for the identification of second phase particles. No scanning or transmission electron microscopy was performed due to time constraints. Investigation by these means would help to resolve questions concerning the effects of processing upon the base matrix. Also further effects of processing on microstructural refinement, such as refinement of grain structures, should be addressed by such microscopy methods.

The aging experiment provided promising results. Upon subsequent solution treatment for one hour, the ductility of both the rolled materials was restored and actually exceeded that of the extruded condition. In addition, the increased strength from the rolling persisted. The increased strength and enhanced ductility persisted throughout the duration of aging at $160^\circ C$. In comparing observed 1% yield strengths it was clearly evident that rolling had enhanced yield strength significantly. Also, it appears that the rolled material attained peak strength in a shorter time period than the extruded material. These same results were evident in comparing ultimate tensile strength, but not to same degree as was evident in the yield strengths. Further comparisons of yield and ultimate strength shows that the rolled material exhibits a much higher yield to ultimate strength ratio. This suggests that the rolled material does not strain harden as much as the extruded material. Finally, the rolled materials also displayed a greater ductility than the extruded condition. This was surprising because with most processing schemes only strength or ductility is enhanced, not both as was the case in this investigation. The mechanical properties displayed at peak strength for all of the processed conditions as well as data for unreinforced 6061 matrix alloy are summarized in Table 6.
Table 6. MECHANICAL PROPERTIES FOLLOWING AGING TO PEAK STRENGTH

<table>
<thead>
<tr>
<th>Material Condition</th>
<th>Yield Strength (MPa)</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Percent Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061-T6 unreinforced</td>
<td>276</td>
<td>310</td>
<td>12</td>
</tr>
<tr>
<td>Extruded</td>
<td>328</td>
<td>350</td>
<td>5.6</td>
</tr>
<tr>
<td>$500^\circ$C Rolled</td>
<td>370</td>
<td>375</td>
<td>7.0</td>
</tr>
<tr>
<td>$350^\circ$C Rolled</td>
<td>375</td>
<td>394</td>
<td>7.0</td>
</tr>
</tbody>
</table>

It has been demonstrated that thermomechanically processing of the material has improved homogeneity of the alumina particle distribution substantially. Further effects of such processing on the microstructure of the matrix have not been addressed in this research. However it is anticipated that refinement of the matrix grain structure has been accomplished. This may include recovered or even recrystallized grain structures. Such structures apparently respond more rapidly to subsequent heat treatment although the responsible mechanisms remain to be determined.

These are significant results, but must be put in proper perspective. Large amounts of strain were necessary ($\varepsilon_{\text{total}} = 5.07$) to homogenize the particle distribution. The question may be asked: Is this the most efficient way to achieve microstructural homogeneity? Improvements in the as-cast particle distribution would seem to offer considerable benefits. Less subsequent mechanical work would be necessary to obtain optimum mechanical properties and thus more complex structural components could be fabricated and yet attain a high level of mechanical properties.

In closing, it would be prudent to note that although the results are noteworthy, they should be viewed as broad comparisons. Tests were limited in number and further testing needs to be accomplished to establish statistically significant results.
Figure 14. Stress-Strain Comparison After Solution Treatment: Ductility is restored for both rolled conditions. Strength increase from the additional strain imparted by rolling persists.
Figure 15. 1% Offset Yield Strength vs. Aging Time: The rolled conditions display considerable improvement of yield strength and attain peak strength at shorter aging time.
Figure 16. Ultimate Tensile Strength vs. Aging Time: Superior strength persists for rolled conditions, but not as dramatically as for the yield strength.
Figure 17. Percent Elongation vs. Aging Time: Ductility of rolled condition is enhanced throughout entire aging process.
V. CONCLUSIONS

1. Due to a non-homogeneous particle distribution the cast condition displayed mechanical properties that were erratic, and characteristic of an extremely brittle material.

2. The extruded condition displayed a more homogeneous microstructure than the cast condition. Ductility was enhanced by extrusion, and a slight decrease in strength was observed.

3. Isothermal rolling of the extruded material at both 500°C and 350°C resulted in a strength increase, but a substantial loss in ductility persisted.

4. Isothermal rolling resulted in a homogenization of the $Al_2O_3$ particle distribution.

5. Particles appeared smaller in the rolled conditions due to spreading of the larger cluster formations observed in the cast and extruded material.

6. Upon subsequent heat treatment (aging at 160°C), strength was increased in the rolled material along with an enhancement in ductility.

7. The rolled material attained peak strength faster than the extruded material.
VI. RECOMMENDATIONS FOR FURTHER STUDY

1. Investigate elevated temperature mechanical properties.
2. Investigate higher volume percent material.
3. Determine the effects of processing upon fatigue and fracture characteristics.
4. Determine the effects of processing upon the base matrix alloy using the Transmission Electron Microscope (TEM).
Figure 18. Stress-Strain Curves for Extruded Material: Slight decrease in strength evident in comparison to cast material. Substantial improvement in ductility observed.
Figure 19. Stress-Strain Curves for 500°C Rolled Material: Slight increase in strength from rolling. Considerable loss of ductility evident.
Figure 20. Stress-Strain Curves for 350°C Rolled Material: Substantial improvement in strength from rolling. Considerable loss of ductility evident.
Figure 21. Stress-Strain Curves for 10 minute (T6) Heat Treatment: Note 500°C rolled material slightly stronger than 350°C rolled material.
Figure 22. Stress-Strain Curves for 30 minute (T6) Heat Treatment: Note 500°C rolled material slightly stronger than 350°C rolled material.
Figure 23. Stress-Strain Curves for 1 hour (T6) Heat Treatment: Substantial strength difference between rolled materials. Suggests 500°C rolled material attained peak strength faster.
Figure 24. Stress-Strain Curves for 4 hour (T6) Heat Treatment: Strength and ductility of rolled material greater than extruded material.
Figure 25. Stress-Strain Curves for 8 hour (T6) Heat Treatment: Increased strength of rolled material persists. Ductility of extruded material greater.
Figure 26. Stress-Strain Curves for 16 hour (T6) Heat Treatment: Strength and ductility of rolled material higher than extruded material. Peak aging time for unreinforced 6061-T6 is 18 hours.
Figure 27. Stress-Strain Curves for 120 hour (T6) Heat Treatment: In overaged condition both strength and ductility of rolled material are superior to the extruded material.
LIST OF REFERENCES


20. Telephone conversation between (Lane, C.), (DURALCAN) and Schaefer T., 20 March 1989.

# INITIAL DISTRIBUTION LIST

<table>
<thead>
<tr>
<th>No.</th>
<th>Copies</th>
<th>Name and Address</th>
</tr>
</thead>
</table>
| 1.  | 2      | Defense Technical Information Center  
       Cameron Station  
       Alexandria, VA 22304-6145 |
| 2.  | 2      | Library, Code 0142  
       Naval Postgraduate School  
       Monterey, CA 93943-5002 |
| 3.  | 1      | Department Chairman, Code ME Hy  
       Department of Mechanical Engineering  
       Naval Postgraduate School  
       Monterey, CA 93943 |
| 4.  | 1      | Naval Engineering Curricular Office, Code 34  
       Naval Postgraduate School  
       Monterey, CA 93943 |
| 5.  | 5      | Professor T.R. McNelley, Code 69Mc  
       Department of Mechanical Engineering  
       Naval Postgraduate School  
       Monterey, CA 93943 |
| 6.  | 3      | Assistant Professor I. Dutta, Code 69Du  
       Department of Mechanical Engineering  
       Naval Postgraduate School  
       Monterey, CA 93943 |
| 7.  | 1      | Mr. William R. Hoover  
       Vice President for Materials Department  
       Dural Aluminum Corporation  
       10326 Roselle Street  
       San Diego, CA 92121 |
| 8.  | 1      | Dr. J.S. Santer  
       Materials Reliability Inc.  
       P.O. BOX 949  
       Naperville, IL 60566 |
| 9.  | 1      | Dr. Lewis E. Sloter, Code AIR 931A  
       Headquarters Naval Air Systems Command  
       Washington D.C. 20361 |
| 10. | 1      | Lt. Thomas A. Schaefer  
       10280 Caminito Rio Branco  
       San Diego, CA 92131 |