Effect of Temperature and Percent Cold Work on the Mechanical Properties of Aluminum Alloy 3104

by

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MECHANICAL PROPERTIES OF ALUMINUM ALLOY 3104

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Committee Chairman: William T. Reynolds, Jr.

Materials Science and Engineering

(ABSTRACT)

The effect of fourth pass cold reduction and final anneal
temperature were investigated for aluminum alloy 3104. The
material was received at 0.019" (82% reduction) and further
reduced to: 84%, 86%, 88%, and 89%. The material was then
heated for 2 hours between 85°C and 160°C.

Samples were uniaxially tensile tested at 0.0167 per
second for yield strength, ultimate strength, and total
percent elongation. Samples showed an increase in ductility
with increasing temperature. This is believed to be the
result of recovery. Prior processing limited the possibility
that age hardening effects would occur. No age hardening was
found. TEM micrographs showed no evidence for the presence of
GP zones or the S' Al₃CuMg metastable phase.
Acknowledgements

Firstly, I wish to thank Dr. Gordon and Dr. Reynolds for their consideration, advice and patience during this "ordeal". Secondly, I wish to thank Victor Dangerfield and Ravenswood Aluminum Co. for supplying the material, real world problem, and help during this study. I am most indebted to Bill Halley for his muscle, brains, and practical experience. I additionally would like to thank the "T-Group": Gang, J.K., Tweed, and Eric for assistance with the TEM and a place to "hang my hat". I also appreciate the assistance Ravi, and Dr. Kander supplied with respect to interfacing the Instron to the Macintosh. I would like to thank Harry Dudley and Bunny McDonald for assistance with sample preparation. Finally, I wish to thank the "heart of the department": Jan, Suzette, Laurie, and Fran for making my graduation possible.
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1. INTRODUCTION

Aluminum alloy 3104 is a manganese-based non-heat treatable alloy, made up of 1.0 weight percent manganese, 1.10 weight percent magnesium, 0.40 weight percent iron, 0.20 weight percent silicon, and 0.16 weight percent copper. This material is used primarily as can stock for the beverage container industry which requires it to have high strength and good formability so as to withstand the drawing and ironing process. The alloy is strengthened by work hardening, solid solution strengthening, and the possible precipitation of copper containing S' phase during final annealing (1).

Drawing and ironing starts with initial cup drawing during which the sheet thickness is reduced by 35 to 40 percent. The cups are then redrawn and their wall thicknesses are reduced through a series of ironing rings, known as ironing, to a final gage of approximately 0.005" (0.127 mm) (2). The can making process requires that the microstructure of the aluminum alloy be controlled so as to produce a fine grain size. The processing of this alloy consists of producing an ingot which is then homogenized, hot rolled, annealed, and cold rolled to allow for retained strength and formability during can making.
The alloy is solidified using the direct chill method which has an average cooling rate of approximately 1°C per second (3). This process produces a microstructure of primary aluminum dendrites with an interdendritic region saturated in Mn, Fe, and Si, and coarse particles composed of Al(Mn,Fe) (4). Approximately 0.75 weight percent Mn, and 0.02 weight percent Fe remain in solid solution after solidification. The remaining Mn and Fe are contained in the approximately 3 volume percent Al(Mg,Fe) particles (4). These coarse (>100 micron) particles can stimulate nucleation, during recrystallization, by supplying areas for acute lattice misorientation, after deformation has taken place.

Following solidification, the ingot is homogenized at approximately 600 °C for 8 hours with a slow cool (≈27°C/hr.) to the hot rolling temperature so as to relieve some of the saturation that remains in the matrix. This slow cool from a high temperature soak insures that the particles made up of Al, Mn, and Fe grow to their maximum size while dispersoids made up of Al, Mn, Si and Fe form from the solid solution and are widely spaced (3). Silicon promotes the precipitation of Fe while Si, Fe, Mg, and Cu promote the precipitation of Mn during homogenization (4).
In a typical homogenized structure, the Mn containing dispersoids will be $\approx 0.25$ microns in diameter with a spacing of $\approx 0.5$ microns. This leaves approximately 0.35 weight percent Mn in solution and reduces the possibility for further precipitation during thermomechanical processing (2). The homogenization treatment also evenly distributes Mg and Cu throughout the matrix.

Hot rolling is performed at temperatures between 500°C and 304°C with an alloy exit thickness of approximately 0.105" (2.67 mm). This process breaks up the as-cast structure and the coarse Al(Mn,Fe) particles and distributes the coarse particles and the fine secondary precipitates through the matrix, while grains are compressed and lengthened. Excess manganese can precipitate during this step (3). Coarse constituents range in size from 0.4 to 18.6 microns with an average size of 3.7 microns and spacing of 24.4 microns after hot rolling (4). Typically, hot rolled secondary precipitates range in size between 0.07 and 0.30 microns with an average size of 0.15 microns and spacing of 0.36 microns (4).

The hot rolled aluminum sheet is then annealed at 350°C for 2 hours with an air cool to fully recrystallize it and precipitate any remaining solute. Recrystallization softens
the material and facilitates further size reduction by cold rolling. It also alters the crystallographic texture.

The material is then cold rolled in several passes to increase the strength of the alloy and to continue uniform particle distribution by metal flow, while grains are further compressed to produce a rolling texture. Cold reduction increases the dislocation density and causes the formation of subgrain structures. Constituent particles range in size from 0.4 to 15.0 microns with a 3.4 micron average and a spacing of 29.6 microns at a sheet reduction of 0.013" (0.33 mm) (4).

Non-heat treatable alloys derive their strength from solutes in solid solution, particles that form during processing, and deformation strengthening resulting from cold rolling. Solid solution strengtheners include Mg, Cu, and Mn. These atoms reside in the metal matrix and are believed to primarily resist dislocation motion through frictional forces (5). Magnesium is an effective solid solution strengthener and is highly soluble in aluminum. This element can, however, form Mg-Si and Al-Mg-Cu second phases during processing. The solid solution strengthening effect of Mn is limited due to low solubility (0.2 to 0.30 weight percent Mn) (3).
Primary and secondary particles increase the strength of the alloy by providing barriers to dislocation motion. Coarse primary and fine secondary particles can assist work hardening by providing sites for dislocation nucleation and multiplication.

Recovery and recrystallization are processes that take place during annealing operations. Recovery is the release of stored deformation strain energy through dislocation rearrangements, such as climb, whereas recrystallization consists of the nucleation and growth of new strain free grains in the matrix. Recrystallization requires that a critical amount of strain is present in the matrix and it is affected by the annealing temperature and heating rate. Higher annealing temperatures decrease the amount of critical strain needed. A fast heating rate decreases the amount of prior recovery leaving more strain energy to drive recrystallization leading to a fine grain size.

Solute atoms impede recovery of the crystal by solute drag. This produces numerous sites of retained strain energy which are potential nuclei for new strain free grains. These nuclei can then grow and produce a fine grain size.

Primary and secondary particles affect
recrystallization in opposite ways. Primary precipitates are coarse, widely spaced particles which can provide sites for particle stimulated nucleation and a fine grain size. Secondary particles, however, can increase the grain size by pinning low angle subgrain boundaries thus reducing the thickness of the boundary walls and the potential sites for nucleation of new grains. These second phase particles also provide a stabilization effect on mechanical properties during partial annealing by helping to control recovery and recrystallization. By controlling the processing steps, the precipitation of particles and work hardening can produce higher strength. This will allow the alloy to keep pace with future demands placed upon it by changes in can production technology.

There is a trend in the industry to produce thinner gauge stock for can making. Higher strength is needed to resist fracture of the material during can production. This also requires that the alloy remain ductile to accommodate the severe deformation placed on it by the drawing and ironing process (67 percent total reduction) (6).

This study attempts to find a peak strength condition without changing the conventional processing. H.D. Merchant et al, studied the effects of strain aging on alloy
3004 and found a peak strength condition at 135°C for warm rolled samples (7). Eiki Usui, et al, found that the strength of alloy 3004 increases to a peak at 200° to 220°C for 20 minutes aging (annealed before 60% cold work) and 150°C for 100 hours (annealed after 60% cold work) (1). This paper presents the effects of final annealing temperature and cold rolling on the ultimate tensile strength, yield strength, and percent elongation for alloy 3104. It also looks at the feasibility of age hardening during the final anneal after the material has been conventionally processed up to that point.

2. EXPERIMENTAL PROCEDURE

Samples were received from Ravenswood Aluminum Company after the third pass of cold rolling at a nominal thickness of 0.019" (0.48 mm). The material was conventionally processed up to this point as described in the introduction. This alloy's composition can be seen in Table 1.

Test sections approximately eight inches long and one inch wide were sheared from the sheet with the long dimension parallel to the rolling direction. These strips were then reduced by: zero, ten, twenty, thirty, and forty
percent (82, 84, 86, 88, and 89 percent reduction, respectively, from the hot rolled thickness) in a single pass using a Stanat Model TA 315 rolling mill which was equipped with rolls five inches in diameter by eight inches in length. The final thicknesses of the samples were approximately: 0.019" (0.48 mm), 0.017" (0.43 mm), 0.015" (0.38 mm), 0.013" (0.33 mm), and 0.0115" (0.29 mm), respectively.

These samples were trimmed to approximately eight inches long by three quarters inch wide and placed in an oil bath for heat treatment. The samples were held for two hours at: 85, 110, 130, 145, and 160 °C. The test strips were then machined into tensile specimens with a two inch gage length.

The test coupons were pulled at a strain rate of 0.0167 per second using an Instron screw-driven mechanical testing machine equipped with a 5,000 newton load cell and interfaced with an Apple Macintosh Computer equipped with Labtech Notebook Software. The computer stored time verses load data in an MS-DOS compatible format using a Dynafiler Disk Drive System. These files were then manipulated using Lotus 123 Software to obtain stress versus strain data which supplied ultimate tensile strength, and 0.2 percent
offset yield strength. Samples were also scribed with gage marks to supply percent elongation data. Four samples at each condition were tested.

Eighty nine percent cold worked samples at room temperature and 160°C were examined using a Phillips EM-420 STEM at 120KV. Three millimeter diameter disks were jet polished using a South Bay Model 500 Jet Thinning Apparatus. Samples were polished in a 2.5 weight percent KI methanol solution at minus 30°C. The samples were polished for approximately 7.5 minutes/side at a pump setting of 1, 90 volts, and 6 mA.

3. RESULTS AND DISCUSSION

Yield strength data are shown in Figures 1-5. These graphs show the change in strength versus final anneal temperature at various percent reductions. Yield strength is shown to remain constant within the ± 1 standard deviation error bars. Further analysis showed no statistical difference in yield strength for each percent cold work.

Ultimate tensile strength data are illustrated in Figures 6-10. These graphs present the change in tensile
strength as anneal temperature changes for the different cold reductions. Figures 6-9 show a trend of constant strengths as the temperature increases for 82, 84, 86, and 88 percent reductions respectively. This suggests that the intermediate anneal temperature (350°C, prior to cold reduction), and the anneal cooling rate (air cool) were not sufficiently high enough to produce a supersaturated solution capable of precipitating GP zones, and the metastable Al-Mg-Cu S' phase. S' is a coherent transitional phase formed from GP zones of Cu and Mg leading to the equilibrium CuMgAl₂ S phase. Usui, et al., showed no age hardening effects for air cooling or solution treatment temperatures below 430°C. (1). The conventional anneal (this study) favors the formation of the equilibrium S phase. This phase represents an over-aged condition.

Figure 10, which shows the tensile strength data for the 89 percent cold worked specimens, illustrates roughly an eight percent increase in strength as anneal temperature is raised. Statistical analysis showed this increase to be insignificant. Figures 16-17 show TEM micrographs for the 89% reduced samples at 20°C and 160°C aging temperatures, respectively. These micrographs show the presence of submicron dispersoids devoid of GP zones which supports the
conclusion that the intermediate anneal was not conducive for age hardening.

Percent elongation data can be seen in Figures 11-15, and show a statistically significant increase in ductility as temperature rises for all reduction conditions examined. This data suggests that recovery in the microstructure is taking place. The average values for yield strength, tensile strength, and percent elongation with respect to change in annealing temperature and percent cold work can be found in the appendix.

4. CONCLUSIONS

Increasing annealing temperature after cold rolling provided insignificant changes in yield and tensile strength for all reductions in sample thickness. Percent elongation data showed significant increases in ductility as annealing temperature increased. This can be accounted for by the recover process.

Precipitation hardening has been shown to occur in a similar alloy in the findings of Usui, et al (1). This hardening effect is believed to occur by the precipitation of GP zones and a metastable Al-Mg-Cu containing S' phase
during final annealing (1).

Precipitation hardening of aluminum alloy 3104 was not observed in the present study. TEM micrographs of the 89% cold rolled samples showed no presence of GP zones or S' phase. The intermediate annealing temperature and cooling rate may be key factors. The present study used material that was annealed at 343°C for 2 hours with an air cool. Usui showed that no age hardening occurred below 430°C or with conventional air cooling (1). Conventional annealing may provide conditions conducive to producing equilibrium S (CuMgAl2) phase due to its low cooling rate.

In conclusion, increasing the intermediate annealing temperature and cooling rate should provide sufficient supersaturation to facilitate precipitation hardening of alloy 3104. The final annealing temperature range should be broadened to increase the driving force for possible S' precipitation. Usui, et al, showed peak strength at \( \approx 200°C \) for solution treatment prior to cold rolling (1). This material's mechanical properties should then be characterized by uniaxial tensile testing, and cup drawing. This drawing process will give insight into the earing behavior of precipitation hardened 3104.
Figure 1: Yield Strength vs. Temp. for 82% Cold Work
Figure 2: Yield Strength vs. Temp. for 84% Cold Work
Figure 3: Yield Strength vs. Temp. for 86% Cold Work
Figure 4: Yield Strength vs. Temp. for 88% Cold Work
Figure 5: Yield Strength vs. Temp. for 89% Cold Work
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Figure 9: U.T.S. vs. Temp. for 88% Cold Work
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Figure 13: Percent Elongation vs. Temp. for 86% Cold Work
Figure 14: Percent Elongation vs. Temp. for 88% Cold Work
Figure 15: Percent Elongation vs. Temp. for 89% Cold Work
Figure 16: TEM Micrograph for 89% Cold Work and 20°C Age
Figure 17: TEM Micrograph for 89% Cold Work and 160°C Age
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REFERENCES


### APPENDIX

#### TABLE 2: MECHANICAL PROPERTIES FOR ALLOY 3104

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