

To the Graduate Council:

I am submitting herewith a thesis written by Tamara Kay McGill-Taylor entitled "Effect of Cold Working and Annealing on Mechanical Properties, Microstructure, and Texturing of Aluminum Alloy 3104." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Metallurgical Engineering.

Charlie R. Brooks

Charlie R. Brooks, Major Professor

We have read this thesis
and recommend its acceptance:

W T Baker

Edward J. Clark

Accepted for the Council:

C. W. Mink

Vice Provost
and Dean of The Graduate School

STATEMENT OF PERMISSION TO USE

In presenting this thesis in partial fulfillment of the requirements for a Master's degree at The University of Tennessee, Knoxville, I agree that the library shall make it available to borrowers under rules of the Library. Brief quotations from this thesis are allowable without special permission, provided that accurate acknowledgement of the source is made.

Permission for extensive quotation from or reproduction of this thesis may be granted by my major professor, or in his absence, by the Head of Interlibrary Services when, in the opinion of either, the proposed use of the material is for scholarly purpose. Any copying or use of the material in this thesis for financial gain shall not be allowed without my written permission.

Signature

Thomas L. Maitland

Date

December 1, 1999

EFFECT OF COLD WORKING AND ANNEALING ON
MECHANICAL PROPERTIES, MICROSTRUCTURE, AND TEXTURING OF
ALUMINUM ALLOY 3104

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Tamara Kay McGill-Taylor

December 1989

DEDICATION

To my father
Bobby L. McGill

ACKNOWLEDGEMENTS

iii

Great appreciation is extended to the many people who assisted me during the course of this research project. In particular, I am indebted to my parents, Mr. and Mrs. Bobby McGill, for their constant support and encouragement, and to my brother, Mike, for assisting me with my computer problems. In addition, I would like to extend deep appreciation to my husband, Greg, for his help and understanding.

I am extremely appreciative to my advisor, Dr. Charlie R. Brooks, for his guidance and support throughout this research project. I also appreciate Dr. W. T. Becker and Dr. E. S. Clark for serving as my committee members.

Special appreciation to Dr. Joseph E. Spruiell, Department Head, for being understanding. Special thanks are extended to the staff of the Material Science and Engineering Department for all their help and support. I am greatly indebted to the machine shop, Doug Fielden, Jack French, Ray Bellamy, Mike Rogers, and Mike Ensor, for their help during this project.

Thanks are extended to the electronic shop, Ted Long, Mike Neal, Steve Stiner, Ron Johnson, and Greg Jones, for their assistance.

I would like to extend appreciation toward Al Carter, Aileen Cagle, and Randy Stooksbury for their help and patience during this research project.

Many thanks are extended to the secretaries of the Material Science and Engineering, Sandy Maples, Sue Turner, Phyllis Klindt, and Chemical Engineering, Sancy Hail, Betty Frazier, for their support. Thanks are extended to Kay Davis and Pat Houser for their help.

Special appreciation is expressed to Ravenswood Aluminum Corporation, Ravenswood, West Virginia for sponsoring this research project.

ABSTRACT

The mechanical properties, microstructure, and texturing as a function of cold working and annealing in aluminum alloy 3104 have been examined. The material used in this research was heavy gauge (>0.150 in.) can stock in the hot rolled condition. A two hour annealing treatment at $332\text{ }^{\circ}\text{C}$ ($630\text{ }^{\circ}\text{F}$) was given in which the heat-up rate was $13\text{ }^{\circ}\text{C}/\text{hour}$ ($25\text{ }^{\circ}\text{F}/\text{hour}$). The material was then cold worked from 12% to 96% reduction. Annealing studies were conducted on 20%, 60%, and 88% cold work materials at six temperatures from $149\text{ }^{\circ}\text{C}$ to $332\text{ }^{\circ}\text{C}$ ($300\text{ }^{\circ}\text{F}$ to $630\text{ }^{\circ}\text{F}$) and for five times ranging from $1/8$ hour to 32 hours.

The tensile mechanical properties, ultimate tensile strength, yield strength, and ductility, showed little variation with sample orientation in sheet samples. Therefore, the material is essentially isotropic (planar) with respect to these properties.

For reductions of 60% and 88%, recrystallization occurred at $316\text{ }^{\circ}\text{C}$ ($600\text{ }^{\circ}\text{F}$) in $1/4$ hour. At 20% cold work, it occurred in 2 hours at $332\text{ }^{\circ}\text{C}$ ($630\text{ }^{\circ}\text{F}$).

During hot rolling, the material had not completely recrystallized and a high fraction cube texture was not present. The intermediate anneal prior to cold working completely recrystallized the material and produced a high cube texture.

Cold working reduced the high fraction of cube texture that was generated from the intermediate anneal. The material with less than 60% cold work showed 90° earing (cube texture) and greater than 60% showed

45° earing (rolling texture). A transition point between the cube and rolling texture was observed at 60% cold work, and no earing occurred.

During annealing, heat-up rates have an effect on the final texture. A fast heat-up anneal increased the cube texture in 20%, 60%, and 88% cold work material. The slow heat-up anneal increased the rolling texture in only the 60% cold work material while having little effect on the texture at 88% reduction.

TABLE OF CONTENTS

CHAPTER	PAGE
1. INTRODUCTION	1
2. LITERATURE REVIEW	3
MATERIAL	3
CASTING	5
HOMOGENIZATION	9
HOT ROLLING	14
RECRYSTALLIZATION	16
TEXTURES (PREFERRED ORIENTATION)	21
3. EXPERIMENTAL PROCEDURE	33
MATERIAL	33
FURNACE CONTROL	33
COLD WORKING AND ANNEALING	35
METALLOGRAPHY AND MICROSCOPY	43
X-RAY DIFFRACTION	48
4. RESULTS AND DISCUSSION	51
MECHANICAL PROPERTIES	51
MICROSTRUCTURES	72
TEXTURING	94
5. CONCLUSIONS AND RECOMMENDATIONS	104
LIST OF REFERENCES	107

CHAPTER	PAGE
APPENDIX	112
VITA	122

LIST OF TABLES

TABLE	PAGE
1. Recrystallization of Al-1.2% Mn in hot rolling [15]	17
2. Chemical composition of aluminum alloy 3104 used in this research.	34
3. Final thickness for percent reduction in cold working.	40
4. ASTM grain size of completely recrystallized material.	95
5. X-ray diffraction results of {200} peak for cold work material.	97
6. Deep drawing results of earing (%) for cold work material	98
7. X-ray diffraction results from tensile specimens of {200} and {220} peaks (normalized) after annealing at 332 °C (550 °F)	99
8. X-ray diffraction results from deep drawn cup specimens of {200} and {220} peaks (normalized) after annealing at 332 °C (550 °F)	101
9. Deep drawing results of earing (%) for annealed material	102

LIST OF FIGURES

FIGURE	PAGE
1. Phase diagram of Aluminum-Manganese system [2]	4
2. Phase diagram of Aluminum-Magnesium system [3]	6
3. Schematic diagram of D. C. process [5].	7
4. Effect of homogenization temperature on the cast structure [8]. HF, 500x.	10
5. Effect of homogenization temperature on the cast structure after soaking [8]. HF, 500x.	12
6. TTT diagram for α formation for alloy 3004 [10].	13
7. Effect of exit temperature on recrystallization of super-purity and commercial purity aluminum. Exit temperature measured 15 seconds after exit from mill [15]	15
8. Relationship between manganese in solid solution and recrystallization [15].	18
9. Conditions of deformation and particle size for which nucleation is observed at particles of Si in rolled aluminum [23] .	20
10. Schematic diagram of the principal microstructural features inside a grain [28]	22
11. Effect of alloying additions on recrystallization in hot-rolling of super-purity aluminum [15].	23
12. Rolling and recrystallization textures in aluminum sheet [31]	25
13. Different types of earing that occur during deep drawing of cups. Rolling direction is vertical [32]	26
14. The recrystallized grain size and the strength of the cube texture component as a function of the parameters given [37] . . .	30
15. Intensity variation of cube and R-textures with rolling temperatures.	31

FIGURE	PAGE
16. Thermocouple locations on aluminum test plate.	36
17. Time-temperature curve of slow heat-up annealing	37
18. Flow diagram of experimental test procedure	38
19. Location of tensile specimens.	41
20. Vertical temperature profile of salt bath.	42
21. Location of metallographic samples in tensile specimens.	44
22. Assembly for mounting metallographic samples.	46
23. Schematic diagram of anodizing technique.	47
24. Ultimate tensile strength at 0°, 45°, and 90° to rolling direction vs. % cold work.	52
25. Yield strength at 0°, 45°, and 90° to rolling direction vs. % cold work.	54
26. Ductility (%) at 0°, 45°, and 90° to rolling direction vs. % cold work.	55
27. Dimple fracture surface (annealed condition) from tensile specimen. Rolling direction perpendicular to fracture surface. 550x	57
28. Ultimate tensile strength in the transverse direction for 20% cold work material at 149 °C, 204 °C, and 246 °C.	58
29. Yield strength in the transverse direction for 20% cold work material at 149 °C, 204 °C, and 246 °C.	59
30. Ductility (%) in the transverse direction for 20% cold work material at 149 °C, 204 °C, and 246 °C.	60
31. Mechanical properties in the rolling direction for 20% cold material annealed at 288 °C (550 °F)	62
32. Mechanical properties in the rolling direction for 20% cold material annealed at 316 °C (600 °F)	63

FIGURE	PAGE
33. Mechanical properties in the rolling direction for 20% cold material annealed at 332 °C (630 °F)	64
34. Ultimate tensile strength in the transverse direction for 60% cold work material at 149 °C, 204 °C, and 246 °C.	66
35. Yield strength in the transverse direction for 60% cold work material at 149 °C, 204 °C, and 246 °C.	67
36. Ductility (%) in the transverse direction for 60% cold work material at 149 °C, 204 °C, and 246 °C.	68
37. Mechanical properties in the rolling direction for 60% cold work material annealed at 288 °C (550 °F).	69
38. Mechanical properties in the rolling direction for 60% cold work material annealed at 316 °C (600 °F).	70
39. Mechanical properties in the rolling direction for 60% cold work material annealed at 332 °C (630 °F).	71
40. Ultimate tensile strength in the transverse direction for 88% cold work material at 149 °C, 204 °C, and 246 °C.	73
41. Yield strength in the transverse direction for 88% cold work material at 149 °C, 204 °C, and 246 °C.	74
42. Ductility (%) in the transverse direction for 88% cold work material at 149 °C, 204 °C, and 246 °C.	75
43. Mechanical properties in the rolling direction for 88% cold work material annealed at 288 °C (550 °F).	76
44. Mechanical properties in the rolling direction for 88% cold work material annealed at 316 °C (600 °F).	77
45. Mechanical properties in the rolling direction for 88% cold work material annealed at 332 °C (630 °F).	78
46. Microstructure at the rolling surface of hot rolled and slow anneal condition. As polished. 200x	79
47. Grain structure in the rolling direction of hot rolled condition. Anodized with Barker's reagent. 200x.	81

FIGURE	PAGE
48. Grain structure in the rolling direction of slow anneal condition. Anodized with Barker's reagent. 200x.	82
49. Grain structure in the rolling direction of 20%, 60%, and 88% cold work. Anodized with Barker's reagent. 200x.	83
50. Grain structure of 20% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.	84
51. Grain structure of 20% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.	85
52. Grain structure of 20% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.	86
53. Grain structure of 60% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.	88
54. Grain structure of 60% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.	89
55. Grain structure of 60% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.	90
56. Grain structure of 88% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.	91
57. Grain structure of 88% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.	92
58. Grain structure of 88% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.	93

CHAPTER 1

INTRODUCTION

An all-aluminum two-piece beverage can was introduced in 1976. The seamless body is produced from a sheet by drawing and ironing, which requires extreme formability. The sheets are produced from directionally cast material by hot rolling, annealing, and cold rolling to the final gauge thickness. Aluminum beverage cans today have a nominal thickness of less than 10 mils. The first alloy produced for beverage cans was 3003 (Al-1%Mn), but was superseded by the stronger alloy 3004 which contains the addition of 1% magnesium.

Aluminum alloy 3104, a variant of 3004, manufactured by Ravenswood Aluminum Corporation is used for can stock in deep drawing of beverage cans. This research project was initiated to evaluate the texture development during their manufacturing process.

The material used for this research was heavy gauge (>0.150 in) can stock in the hot rolled condition. Ravenswood has been unsuccessful in producing acceptable final sheet product from this stock. Scrapping the material is required, which is costly. A complete evaluation of their processing techniques was necessary to determine if this material could be salvaged into an acceptable final sheet product.

To produce a suitable final product, control of grain size and texturing (easing) is necessary. Therefore, close control of composition, fabrication conditions, and microstructural changes at all processing stages is required.

The mechanical properties as a function of percent cold work after slow annealing will be used in cold mill preset calculations. These will be used to effectively set rolling equipment in order to optimize reduction and speed with minimum down times.

The 20%, 60%, and 88% cold work materials were annealed at various temperatures and times to examine the changes in mechanical properties and microstructures. The data will be integrated for time/temperature annealing response of automated furnace cycles by Ravenswood.

An unexceptable flaw in deep drawing of beverage cans is the formation of ears. These ears are wavy edges around the circumference of the cup forming at different locations depending upon predominating texture. Customer demands have produced close tolerances on acceptable easing. Thus, a complete understanding of texture development between cold rolling and recrystallization is needed.

CHAPTER 2

LITERATURE REVIEW

Recrystallization and texturing of materials is covered extensively throughout the literature. However, this literature review is restricted to aspects of relevance to current processing techniques used by Ravenswood Aluminum Corporation.

MATERIAL

The International Alloy Designation System (IADS), in 1970, gave each wrought aluminum alloy a four digit number [1]. This established a standardized format for designating and selecting aluminum alloys. The first number indicates the major alloying element. The 3XXX series refers to the aluminum-manganese (Al-Mn) or aluminum-manganese-magnesium (Al-Mn-Mg) alloys. These alloys contain over 1 wt. % manganese, and some alloys have up to 1 wt. % magnesium.

Manganese is added primarily to increase strength and hardness. From the Al-Mn phase diagram in Figure 1, the maximum solubility of manganese in aluminum is 1.82 wt. % at the eutectic temperature of 658 °C (1216 °F). However, iron as an impurity within aluminum reduces the solubility of manganese to 1.25 wt. % [3]. Also, iron and silicon (as an impurity) combine with manganese to form large primary particles (AlMn_6)

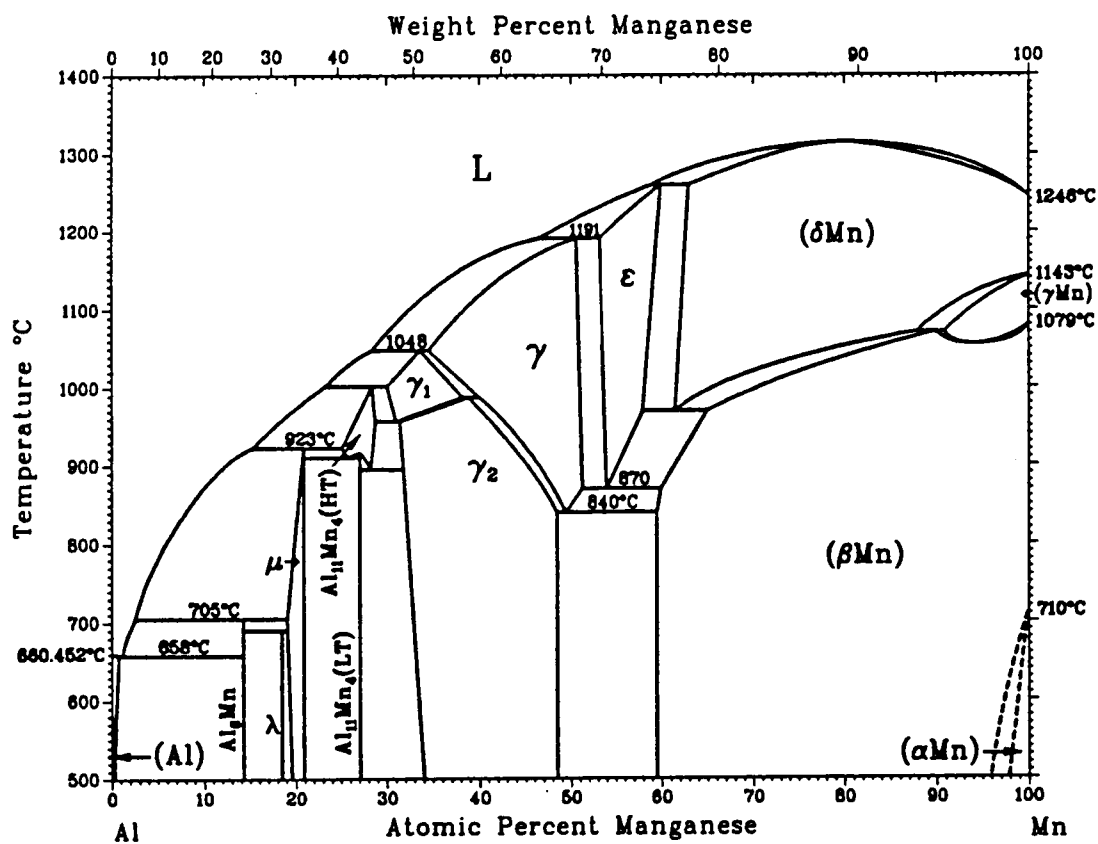


Figure 1. Phase diagram of Aluminum-Manganese system [2].

during casting. High amounts of manganese increase the amount of the large intermetallic particles, resulting in lower ductility.

In aluminum alloy 3104, magnesium is added to increase the degree of strain hardening, and hence these alloys have increased strength over the Al-Mn alloys. At 25 °C, the solubility of magnesium in aluminum is < 1.15 wt. % as shown in Figure 2.

Aluminum alloy 3104 is produced primarily in sheet form. It has increased strength, good ductility, and excellent corrosion resistance which provide a good material for the manufacturing of beverage cans [3].

The 3XXX series of aluminum alloys are classified as non-heat-treatable. In non-heat-treatable alloys, the strength is increased primarily through work hardening, i.e., cold working.

CASTING

The conventional way of producing wrought aluminum alloys is by direct-chill (DC) semi-continuous method as shown in Figure 3. The DC process involves introducing molten aluminum metal into a watercooled mold. Solidification occurs initially at the mold-metal interface which is lowered once a coherent solidified shell has formed [5]. The size and shape of the two-phase region of the ingot (liquid + solid) depends on the temperature of the liquid metal, casting speed, heat removal rate, and also on the alloy being cast [5]. Variations in thermal history affect the formability of Al-Mn alloys [6].

The as-cast ingot retains excess manganese in solid solution resulting in a supersaturated solid solution. A higher amount of manganese in

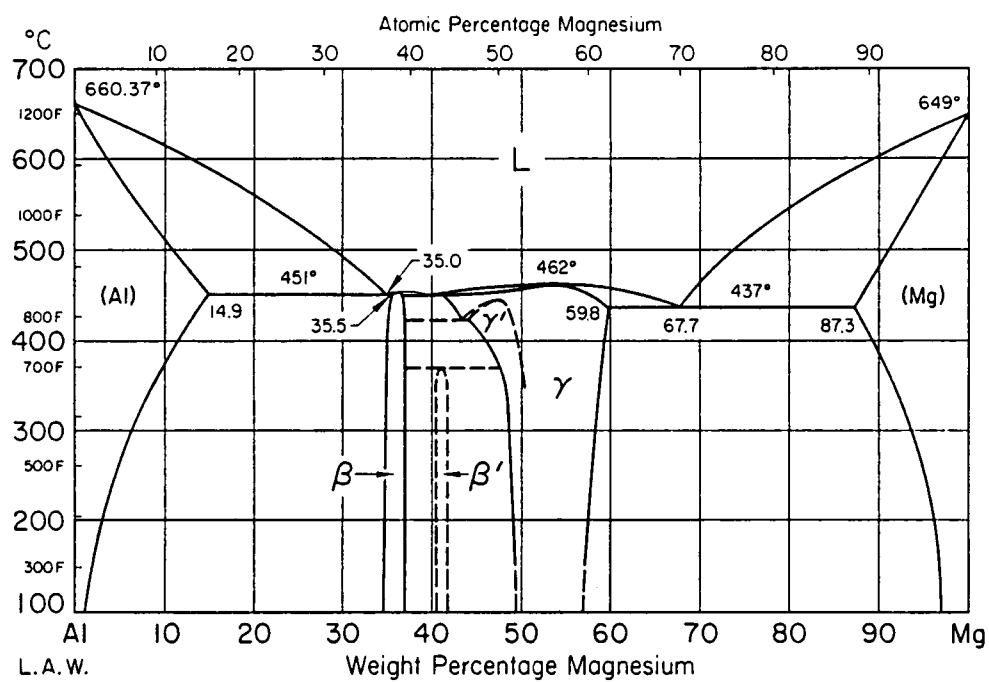


Figure 2. Phase diagram of Aluminum-Magnesium system [3].

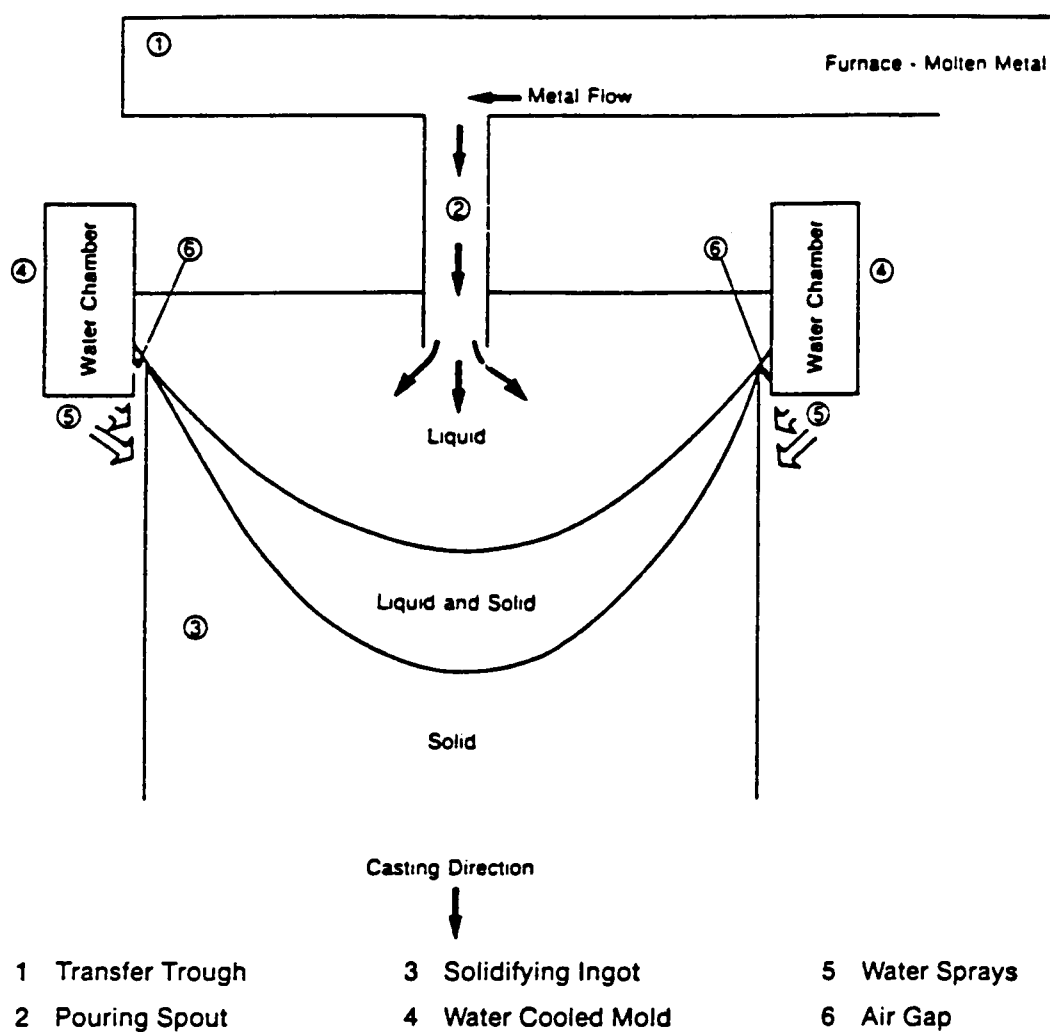
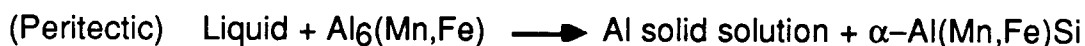


Figure 3. Schematic diagram of D. C. process [5].

supersaturation can occur if higher freezing temperatures are used during casting. The two phases formed during casting are primarily $\text{Al}_6(\text{Mn,Fe})$ and some $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$. About 80-90% of the primary particles are the $\text{Al}_6(\text{Mn,Fe})$ orthorhombic phase while 10-20% are the $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$ cubic phase [7, 8, 9]. The type and amount of each phase depends on the alloy composition and freezing rate during casting. The $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$ particles primarily form during casting from the eutectic reaction, and the remainder occurs by the peritectic reaction from the $\text{Al}_6(\text{Mn,Fe})$ particles. Higher freezing rates and a combination of higher magnesium and lower silicon content than normal suppresses the α -phase formation in casting. This is due to silicon tied up as Mg_2Si . In this case, the peritectic reaction is controlled by the disassociation of Mg_2Si [10]. Below are the eutectic and peritectic reactions:

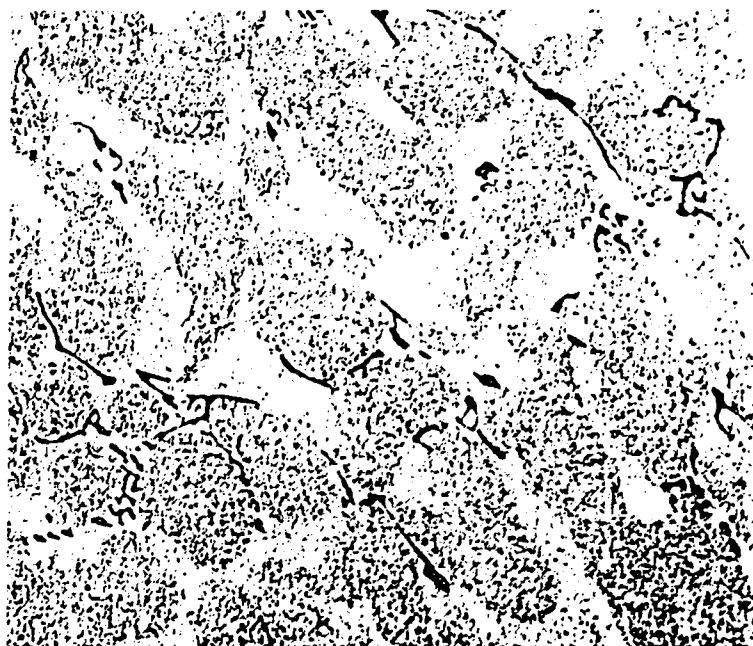


Subsequent heating (homogenization) results in the transformation of $\text{Al}_6(\text{Mn,Fe})$ to $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$. This will continue until equilibrium proportions of $\text{Al}_6(\text{Mn,Fe})$ and $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$ are satisfied. The amount of transformation is discussed in the next section.

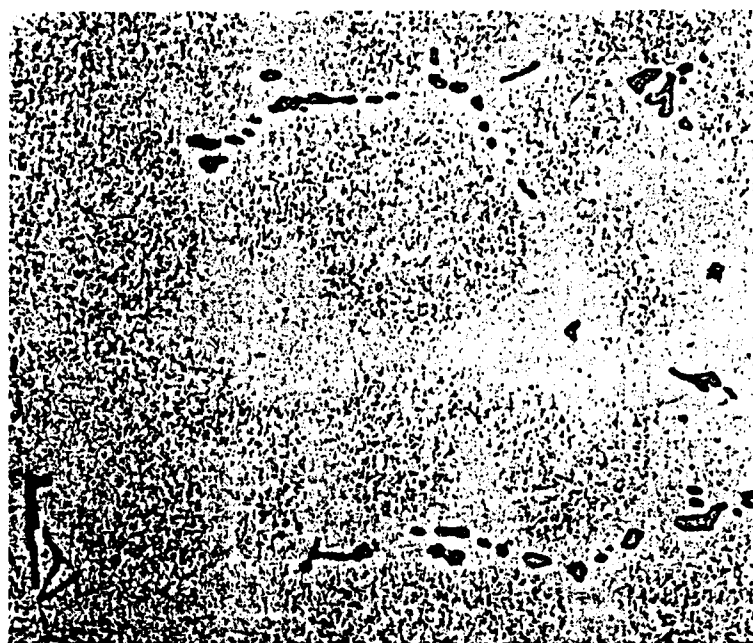
HOMOGENIZATION

The first step after casting is usually homogenization. Homogenization reduces the microsegregation by promoting diffusion of solute atoms within the grains [5] through the process of thermal exposure [10]. Homogenization temperatures [10] of 530-620 °C (986-1148 °F) for 3XXX series alloy are near the solidus temperature [10] of 630-645 °C (1166-1193 °F). Batch-type pit furnaces are generally used with heating by electricity, direct or indirect gas firing, or by fuel, indirect fired, all with air-convection heat transfer [11]. The maximum ingot length in a vertical pit furnace is limited by the amount of distortion an ingot can withstand under its own weight at high temperatures. Homogenization and preheating prior to rolling (soaking) can be combined into one process. The ingot will be held for predetermined time at the homogenization temperature then lowered to the temperature required for hot rolling. Homogenization can have a profound effect on grain size, earing, and mechanical properties that develop upon subsequent hot rolling, annealing, and cold rolling [12]. Thus, homogenization impacts the properties of the final product [10].

A study by Westerman and Smith [8] showed the influence of homogenization and soaking temperatures on Mn-rich particle distribution. They found that higher homogenization temperatures, near the solidus temperature, left larger amount of manganese in solid solution. This is due to the dissolution of the dispersoids and primary particles. Also coarsening (or spheroidizing) occurs, as shown in Figure 4. After homogenization, Ravenswood Aluminum Corporation soaks the ingots at 454 °C (850 °F) prior to hot rolling. This time can vary up to 128 hours. The density of the fine



a) 538 °C (1000 °F)



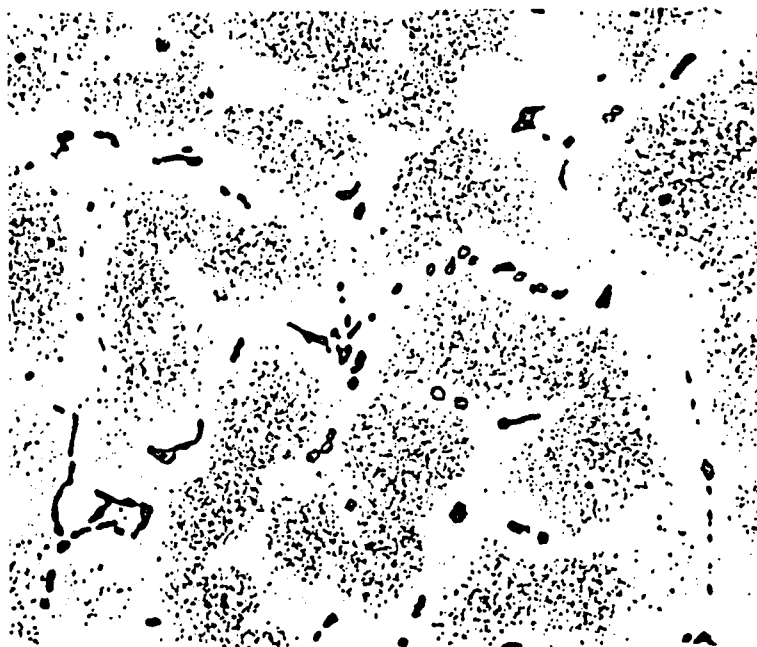
b) 616 °C (1140 °F)

Figure 4. Effect of homogenization temperature on the cast structure [8].
HF, 500x.

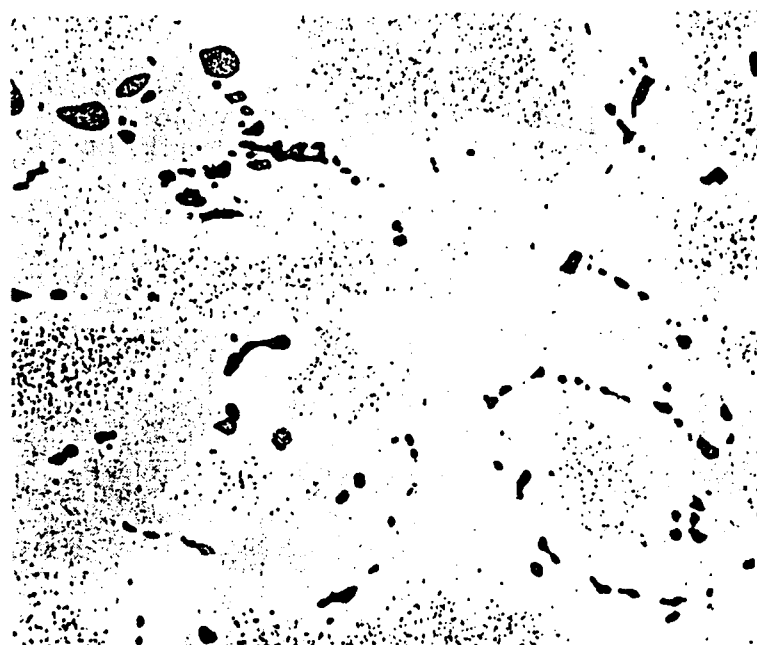
dispersoids precipitated after soaking is less at higher temperatures (Figure 5). Increasing the preheating temperature has an effect on the size and distribution of both the primary and dispersoid particles, which then affects the degree of recrystallization after hot rolling. Higher temperatures coarsen the large primary particles with wide separations and hence produce a less dense distribution of dispersoid particles.

In Al-1.25% Mn alloy, preheating temperature and time are contributors to earing. Increases in soaking time prior to hot rolling can cause small increases in 45° earing [13]. This was also found by Ravenswood [8]. Increase in soaking time (up to 128 hours) increases the dispersoid density and inhibits the recrystallization of cube oriented grains.

During homogenization, the percentage of $\text{Al}_6(\text{Mn,Fe})$ decreases while that of $\alpha\text{-Al}_{12}(\text{Mn,Fe})_3\text{Si}$ increases. This is due to the diffusion of Mn and Si to the $\text{Al}_6(\text{Mn,Fe})$ particles which transforms into $\alpha\text{-Al}(\text{Mn,Fe})\text{Si}$. The amount of transformation depends upon the homogenization temperature and silicon content [8, 10, 14]. Increase in silicon (0.3 wt. %) and high homogenization temperatures cause 100% transformation to the α -particles [8, 10]. From the time-temperature-transformation (TTT) diagram in Figure 6, this effect is shown. The data from Westerman [8] agree with Merchant [10] for 100% transformation at 570 °C (1058 °F) in high silicon content alloys. The reaction depletes the manganese from the Al matrix up to several mm around the coarse particles [7].

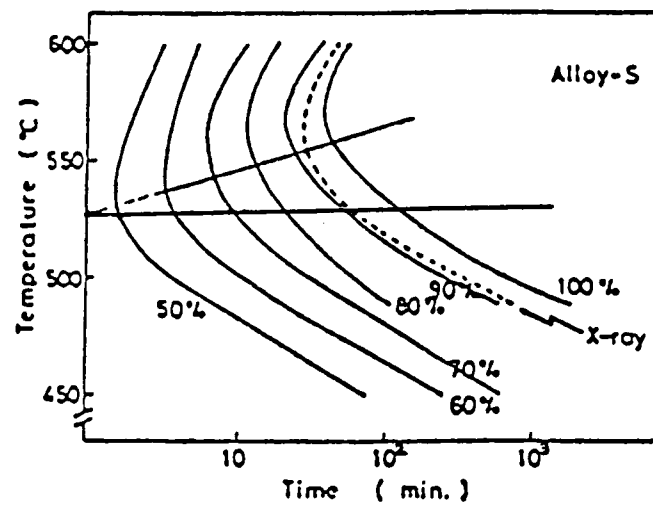


a) Homogenized at 616 °C (1140 °F), soaked at 454 °C (850 °F) for 8 hours

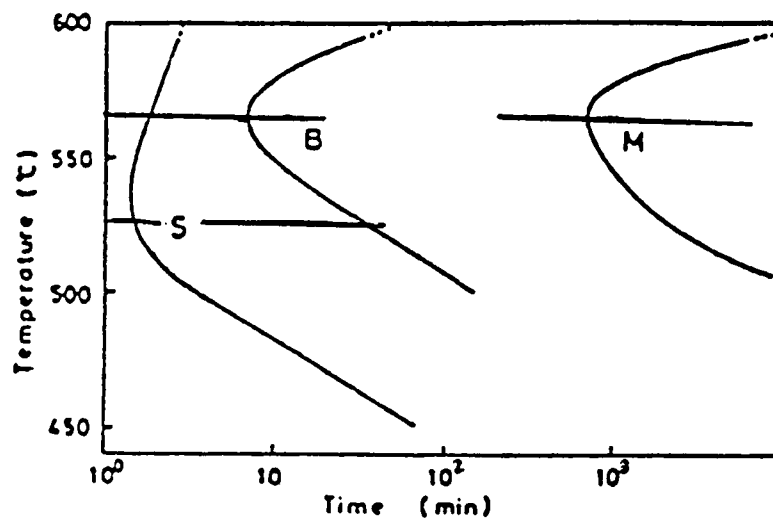


b) Homogenized at 621 °C (1160 °F), soaked at 454 °C (850 °F) for 8 hours

Figure 5. Effect of homogenization temperature on the cast structure after soaking [8]. HF, 500x.



a) Complete transformation for high silicon content (0.35%)



b) 50% transformation c-curves: Alloy B-0.20% silicon, Alloy S-0.35 % silicon, and Alloy M-1.6% Mg

Figure 6. TTT diagram for α formation for alloy 3004 [10].

HOT ROLLING

Blade's [15] study on the effect of hot rolling temperatures of super purity aluminum showed that recrystallization occurred primarily after rolling, not during the actual deformation. Figure 7 shows that the amount of recrystallization after hot rolling is a function of exit temperature. Generally, dynamic recrystallization [16] during hot rolling in Al-Mn alloys does not occur, but dynamic recovery occurs easily to form a well-defined subgrain structure [17, 18, 19]. During recovery, subgrain boundaries are formed from the rearrangement of dislocations. Hollinshead and Sheppard [20] observed from transmission electron microscopy (TEM) that the subgrain size formed during hot rolling increased at higher deformation temperatures. The increase in recovery rates at higher temperatures is due to the ease and rapid rearrangement of dislocations. At lower deformation temperatures, the small subgrains are less defined and have higher dislocation densities [20].

Partial recrystallization may occur during delays between hot rolling passes [7]. The amount of recrystallization will decrease from the surface inward toward the center of the sheet. If during this partial recrystallization there is an increase in cube oriented grains, a decrease in the deformation texture (45° earing) occurs by the growth of new grains of the recrystallized cube texture (90° earing). If hot rolling techniques produce partial recrystallization, a variable structure will form. This variability can cause inconsistent earing behavior [15] due to the variation in transformation from a deformation texture to a recrystallized texture containing either retained rolling or cube texture.

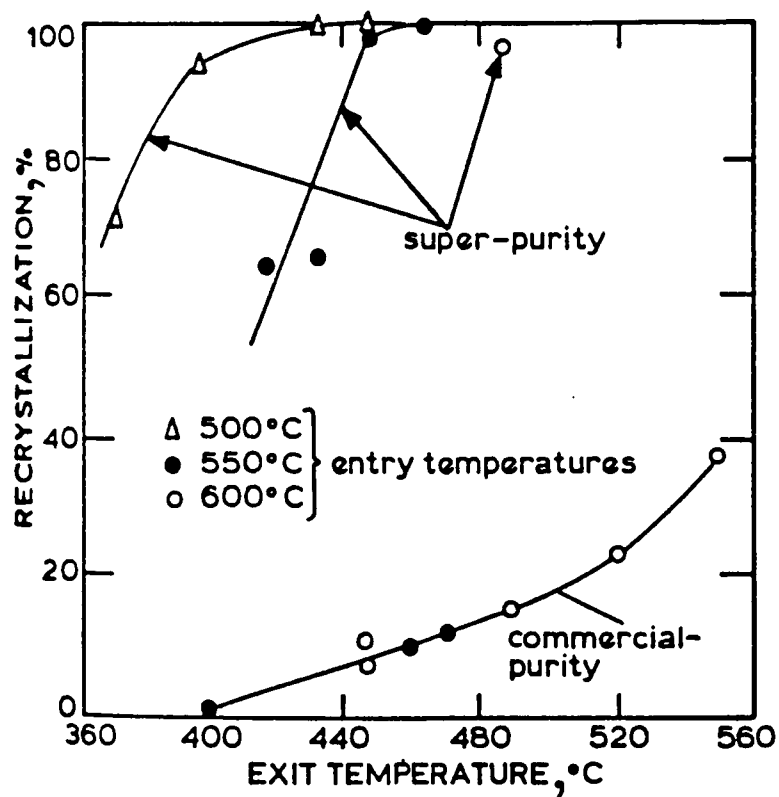


Figure 7. Effect of exit temperature on recrystallization of super-purity and commercial purity aluminum. Exit temperature measured 15 seconds after exit from mill [15].

Table 1 shows the percent recrystallization of an Al-1.2%Mn alloy after hot rolling. The homogenization temperature affects the amount of recrystallization after hot rolling due to the amount of fine precipitates and the amount of manganese left in solid solution [15]. Decreases in both the amount of fine precipitates and of Mn in solid solution increased the percent recrystallization after hot rolling. Blade reported no recrystallization occurred during rolling around 600 °C (316 °F) (in the solid solution region) if the manganese in solid solution exceeded 0.8 wt % (Figure 8).

RECRYSTALLIZATION

Recrystallization is the nucleation of strain-free grains within the cold-worked matrix and subsequent growth of these grains [21]. The temperature and time for recrystallization depends on the material and the amount of prior deformation (among other factors). The recrystallization temperature is generally greater than $0.4T_m$, where T_m is the melting point, absolute. Increasing cold work increases the dislocation density resulting in more nucleation sites for recrystallization, therefore lowering the recrystallization temperature.

The recrystallization process in solid solution alloys occurs at the grain boundaries or in the deformation zones at heavier reductions [16]. The solid solubility at ambient temperature of the majority of alloying elements in aluminum is very limited. These alloys, including Al-Mn alloys, contain two phase regions where intermetallic particles are in the solid solution matrix. The recrystallization kinetics of these two phase regions becomes very complex. Recent studies [7, 18, 22, 23, 24, 25, 26] of the influences of

Table 1. Recrystallization of Al-1.2 % Mn in hot rolling [15].

Heat-treatment	* Recrystallization, %					
	Hot rolled at 600 °C			Hot rolled at 625 °C		
	Rolling reductions			Rolling reductions		
	40%	50%	60%	40%	50%	60%
As-cast	2	4	10	1	7	12
Homogenized at 16 h/625 °C	12	33	40	28	31	37
Homogenized at 16 h/550 °C	2	7	15	1	4	6

* Average of 3 specimens.

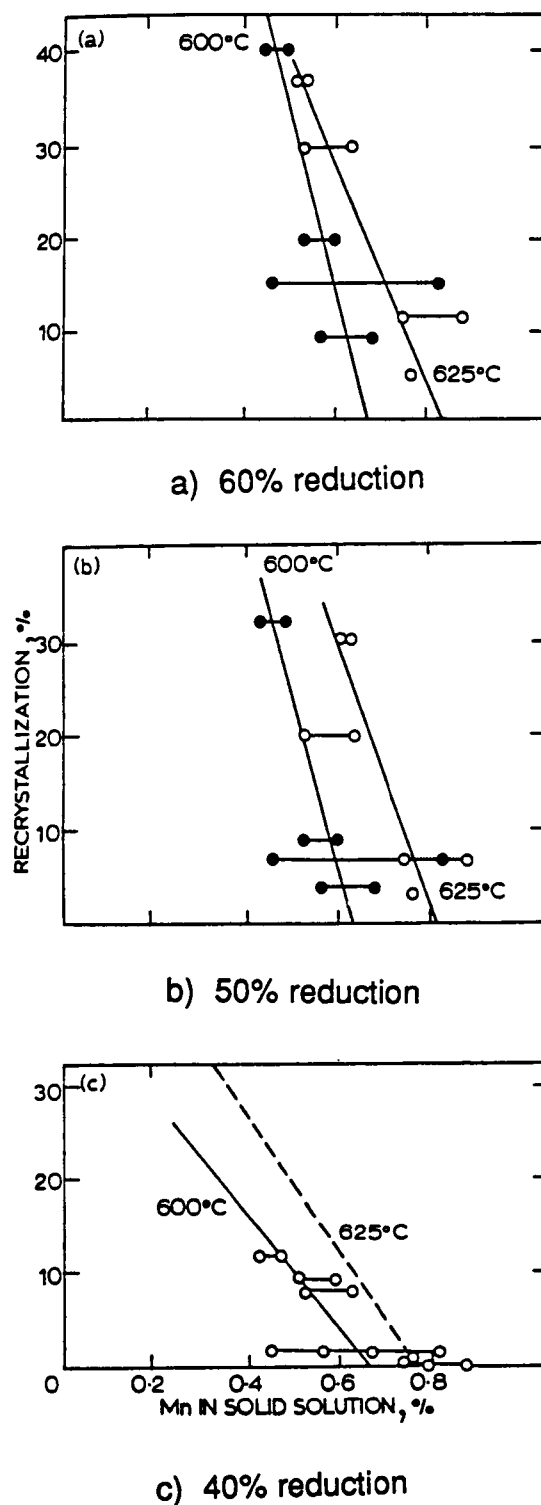


Figure 8. Relationship between manganese in solid solution and recrystallization [15].

intermetallic particles show that particle size and distribution can act as either recrystallization promoters or inhibitors. It has been suggested [7, 16, 18, 23, 24, 25, 27, 28] that widely spaced coarse particles (> 1 mm diameter) promote recrystallization by acting as nucleation sites. Conversely, the smaller, closely spaced particles inhibit recrystallization.

Humphrey [23, 29] identified that recrystallization is retarded or inhibited by closely dispersed particles. These particles can cause a pinning of subgrain boundaries, and thus inhibit recrystallization. Lloyd [22] observed that Mn precipitates (fine dispersoids) prevent migration of the substructure and hence retard recrystallization. The recrystallization process starts at the precipitation-free zones formed during casting operations. Due to reduced sites of nucleation from dispersoids, extensive growth of a few nuclei produce coarse grains.

Several investigators [7, 26] conclude that recrystallization in Al-Mn alloys starts at or near the coarse primary particles. During deformation, the large, non-deformable particles cause dislocation pile-ups, i.e., increased dislocation densities. Nucleation sites for recrystallization occur primarily at dislocations or subgrain boundaries. Humphrey [23, 27] found that recrystallization originates within the deformation zone at pre-existing subgrains, but the subgrains are not always located at the particle/matrix interface. By rapid sub-boundary migration [23], the subgrain grows until the deformation zone is consumed. Figure 9 shows that with greater amounts of reduction smaller particle diameters are needed for recrystallization nuclei. The final grain size in particle stimulate nucleation is a function of interparticle spacing [27].

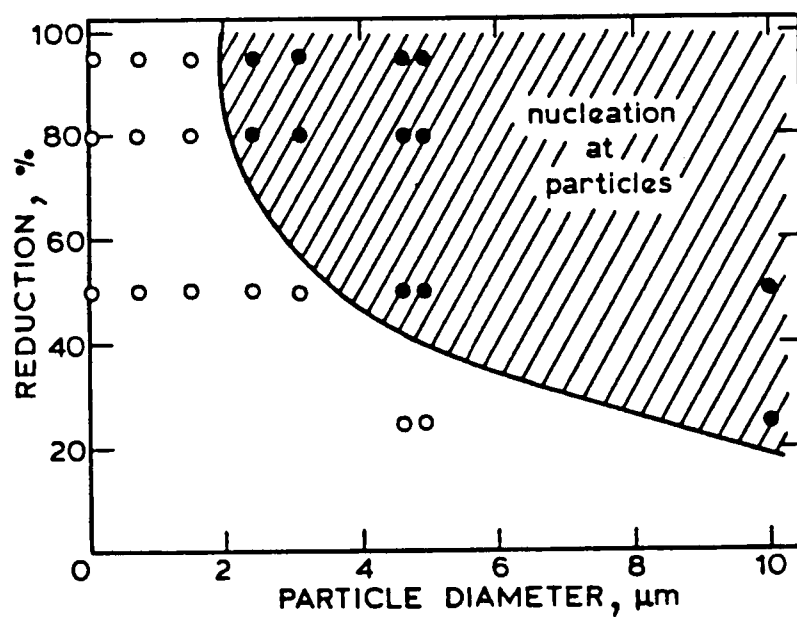


Figure 9. Conditions of deformation and particle size for which nucleation is observed at particles of Si in rolled aluminum [23].

The Al-Mn alloys form a bimodal particle distribution [28] containing a mixed particle structure: coarse distribution of large inclusions formed during casting and a fine particle dispersion formed during any subsequent annealing (Figure 10). Iron and manganese are strong recrystallization inhibitors. Figure 11 shows the effect of alloying elements on recrystallization after hot rolling. Iron content in Al-Mn alloys produces large intermetallic particles which accelerates recrystallization, lowers the recrystallization temperature, and reduces the solubility of manganese.

An increase in manganese from 0.25 wt. % to 0.5-1.0 wt % in aluminum alloys changes the nucleation site of recrystallization [16]. At lower Mn content, recrystallization starts primarily in deformation zones, whereas in higher Mn alloys recrystallization was observed in regions near the coarse intermetallic particles. These particles have an influence on the recrystallization temperature and subsequent grain size. Increasing volume fraction [16] of the large primary particles, with constant composition, significantly reduced the recrystallization temperature and produced a finer grain size.

TEXTURES (PREFERRED ORIENTATION)

Texture is due to the orientation of grains in a preferred crystallographic direction. This frequently results in a material with anisotropic behavior. Texture can form during casting, plastic deformation, and annealing [30]. By determining the effect of processing variables, texture can be controlled.

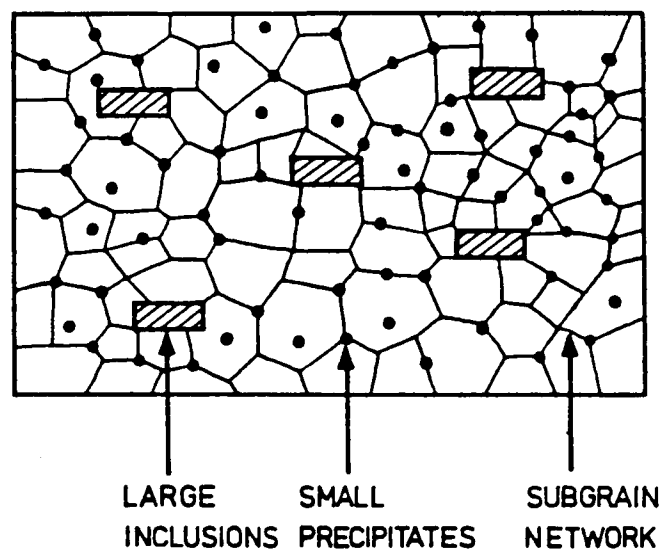


Figure 10. Schematic diagram of the principal microstructural features inside a grain [28].

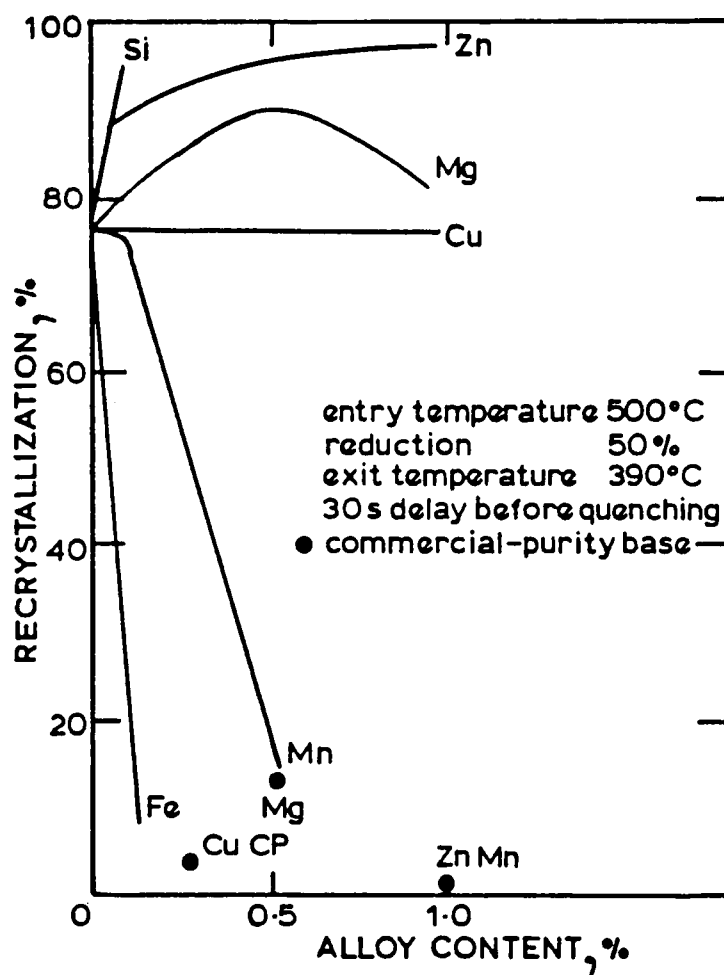


Figure 11. Effect of alloying additions on recrystallization in hot-rolling of super-purity aluminum [15].

Upon casting, the grain structure may be equiaxial and free of crystallographic preferred orientations. When columnar grains form during casting, the grains form a texture by alignment in the $\langle 100 \rangle$ direction [30]. During plastic deformation of sheets by rolling, the grains in aluminum initially become aligned in the $\{110\}\langle 112 \rangle$. The $\{110\}$ plane is parallel to the rolling surface with the $\langle 112 \rangle$ direction parallel to the rolling direction. If extensive cross-slip occurs, other rolling textures are produced which have been described as an orientation spread around the $\{112\}\langle 111 \rangle$ [31, 32]. Figure 12 shows the two primary crystallographic orientations of rolling (deformation) textures in aluminum sheets.

After rolling, the material is generally annealed. The recrystallization process can produce new grains with a different orientation than the deformed texture. In aluminum, the recrystallized texture can be the $\{100\}$ plane oriented parallel to the rolling surface and the $\langle 001 \rangle$ direction in the rolling direction [30]. This is also referred as the cube texture (Figure 12). Upon annealing, some recrystallized grains may retain the rolling texture. This texture is referred as the R- texture, where R- is the retained rolling texture. The rolling texture has been described as an orientation aligned near $\{123\}\langle 412 \rangle$.

The formation of crystallographic textures is important in forming operations of aluminum two-piece beverage cans. A common, unacceptable flaw in sheet materials is the formation of ears during deep drawing. Ears are wavy edges around the circumference of a cup. Figure 13 shows different types of ears. Ears show pronounced directionality in a sheet, and form

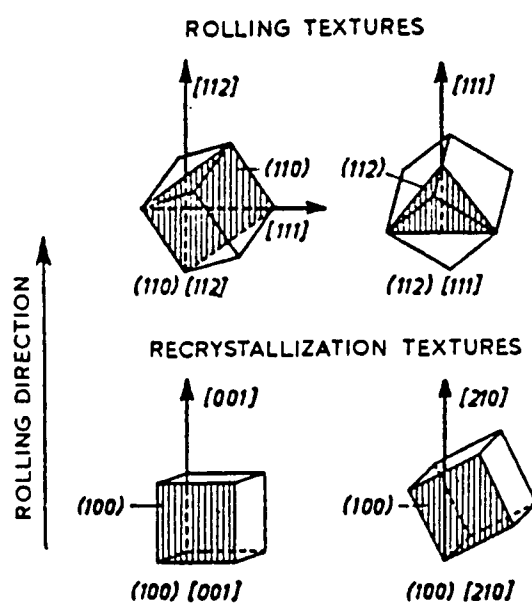


Figure 12. Rolling and recrystallization textures in aluminum sheet [31].



a) Rolling texture - 45° earring



b) Recrystallization texture - 90° earring



c) Mixed texture - 45° and 90° earring



d) Random texture - no earring

Figure 13. Different types of earring that occur during deep drawing of cups. Rolling direction is vertical [32].

during deep drawing at varying positions depending upon the predominating texture. Below are the ear locations due to different textures:

- 1) Rolling texture: Four ears at 45° to rolling direction. Figure 13 (a).
- 2) Recrystallization (annealing) texture: Two ears at 0° to rolling plus two ears at 90° to rolling direction. Figure 13 (b).
- 3) Mixture of Rolling and Recrystallization texture: Eight (total) small ears at 0° , 45° , and 90° to rolling direction. Figure 13 (c).
- 4) Balance of Rolling and Recrystallization texture: No ears. Figure 13 (d).

Large ears cause an additional trimming operation, larger blanks, and failures during the drawing operation by splitting in the troughs between the ears. The area of the material that forms the ears is drawn faster than the material in the troughs.

Earing control in aluminum alloy is achieved by balancing the 90° earing and the 45° earing to yield an effectively isotropic material. The amount of cube texture needed to produce this effect in this material is about 20% with the remainder being the retained rolling texture.

There are several investigations [17, 25, 33, 34, 35, 36, 37, 38] in the literature on the influence of second phase particles on the texture development. Orsund and Nes [35] found that large primary particles were surrounded by deformation zones and recrystallization occurred preferentially in these zones. The subgrains within the deformation zone surrounded the large primary particles. These subgrains, acting as recrystallization nuclei, will generally have an orientation similar to the matrix subgrains [40]. This results in growth of the new grains in the same

orientation as the deformed matrix retaining the rolling texture. Anderson and Nes [33] observed that grains growing from deformation zones contributed to a R- texture. At the onset of recrystallization, cube oriented grains initially show a higher rate of nucleation, but the rate decreases, indicating that there are a limited number of nuclei available during recrystallization [34, 39]. This indicates that the nucleation and growth of R- and cube-nuclei are different [39]. The dispersoid content can affect the strength of the cube texture [38]. Increasing dispersoid density retards recrystallization of the R- nuclei, therefore allowing the cube oriented grains to grow preferentially. According to Nes [40], the fine dispersoid particles promote the cube texture by increasing the cube component in DC cast Al-Mn alloys.

Blade [17] suggests that the cube oriented growth of grains is favored more than the retention of the deformation textures. Therefore, any factor that can restrict the nucleation or growth will influence the cube texture formation and on subsequent earing behavior. This effect on nucleation and growth would be a finely dispersed second phase [17].

Orsund and Nes [36] observed that the annealing temperature changed the nucleation site for recrystallization in Al-Mn alloys with large primary particles. At high annealing temperatures, nucleation of new grains occurred in the deformation zone resulting in a random texture. At lower annealing temperatures, subgrains from the matrix grew into the deformation zone and consumed it. This resulted in a near rolling texture (R- texture).

Contrary to the literature, Westerman and Smith [8] of Ravenswood Aluminum Corporation reported that the large primary particles increased the

90° earing behavior. The literature [35, 36, 39] states that large primary particles reduce 90° earing, but finely dispersed particles enhance 90° earing. Figure 14 shows the relationship of the large particles and dispersoids on grain size and cube texture. Particle stimulated nucleation of recrystallization indicates that the deformation zones around the primary particles do not have preferred orientations [34].

Too large primary particles can have a detrimental effect on the formability during deep drawing operations [34]. However, the size of the large primary particles that would cause problems was not stated.

Cold rolling temperatures have an effect on the formation of cube oriented grains. Rida and Hutchinson [41] found that rolling copper at lower temperatures increased shear band formation. These shear bands eliminated the formation of a strong cube texture and randomly oriented grains nucleated in the shear bands during annealing. The study by Dons and Nes [42] on aluminum could not detect a difference in shear band density and rolling temperatures. But they [42] did observe a change in the intensity of the cube and R- textures with rolling temperature. Figure 15 shows that the cube/R texture ratio increased with increasing rolling temperature. This resulted in an increase in cube oriented grains on subsequent annealing.

Texturing is normally determined through the generation of x-ray pole figures. Our study consists of measuring the relative x-ray integrated intensities normal to the rolling plane. The relative intensities were obtained by normalizing against a randomly oriented powder standard. The x-ray data

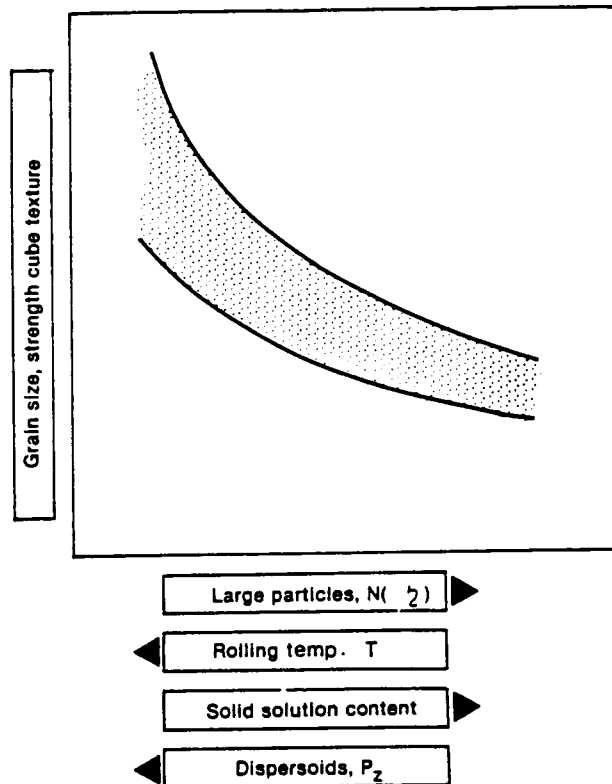
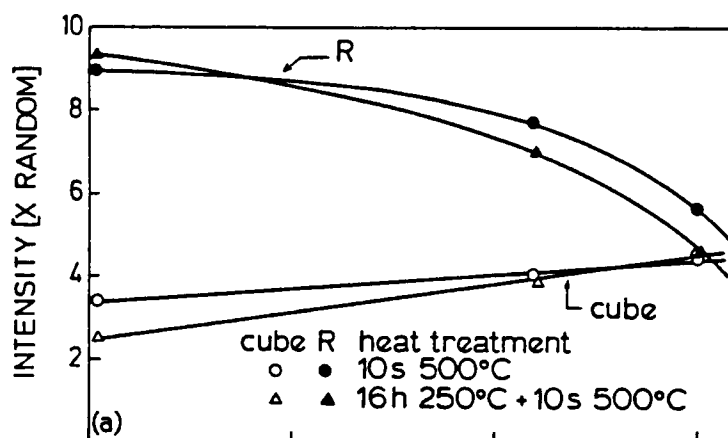
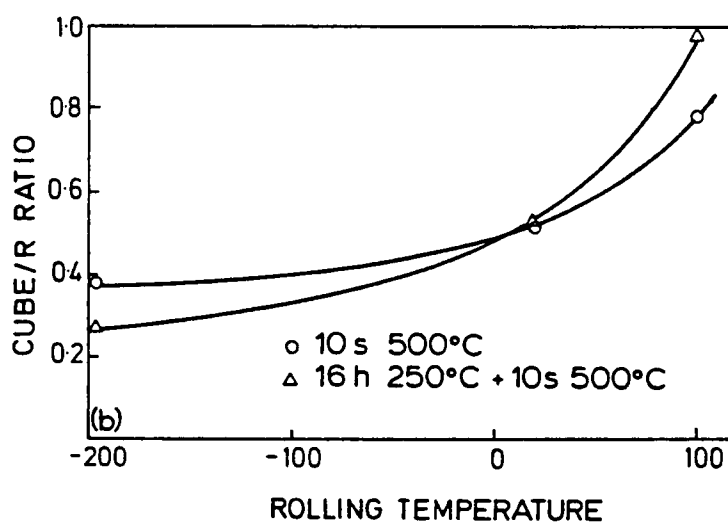


Figure 14. The recrystallized grain size and the strength of the cube texture component as a function of the parameters given [38].



a) cube and R pole figure maxima



b) cube/R ratio

Figure 15. Intensity variation of cube and R- textures with rolling temperatures [42].

will be used to determine if a correlation between the relative intensities and the deep drawn cups of the same condition exist.

CHAPTER 3

EXPERIMENTAL PROCEDURE

MATERIAL

The chemical composition for aluminum alloy 3104 used in this research is listed in Table 2. The fabrication process from the preheat cast ingot to its final sheet form was by hot rolling at 449 - 493°C (840 - 920°F) to a thickness of 0.503 cm (0.198 in) and air cooled. This is the as-received condition from Ravenswood Aluminum Corporation. The sheets were approximately 35.4 cm x 135 cm (14 in x 53 in) and labeled A through H by random selection for documentation purposes. The sheets were sheared into smaller sections approximately 31.8 cm (12.5 in) wide for annealing.

FURNACE CONTROL

A programmable temperature controller (Omega Series CN-2010) was used to control the furnace temperature within $\pm 1^{\circ}\text{C}$ ($\pm 2^{\circ}\text{F}$) and to program the slow heat-up rate of 13.9°C/hour (25°F/hour) to 332°C (630°F) and held for 2 hours. An Omega Model #RD-2062 chart recorder with a thermocouple module was used to record the furnace temperature. The controller was tuned by adjusting the proportional band (PB), reset, and rate values. See Appendix for details of the parameter set-up. The final settings for controlling

Table 2. Chemical composition of aluminum alloy 3104 used in this research.

Element	Weight %
Silicon	0.23
Iron	0.43
Copper	0.20
Manganese	1.00
Magnesium	1.15
Aluminum	96.99

the furnace temperature was PB = 39%, reset = 0.40 resets/minute, and rate = 0.

Initially, a K-type thermocouple inside a stainless steel sleeve was used to profile the furnace. Large variations in temperatures were observed. An Al sheet 27.9 cm x 40.6 cm x 0.64 cm (11 in x 16 in x 0.25 in) with nine thermocouple connections was used to simulate the sheet heat-up during annealing. Figure 16 shows the locations. K-type 24 gauge Duplex insulated thermocouple wire was used. The thermocouple ends were welded together by tungsten inert gas (TIG). The hole depth for the thermocouple connection was approximately 2-3 times the largest diameter weld bead. The thermocouples were connected to the sheet by peening the hole closed. An Omega 10 point rotary selector switch was connected to the thermocouple wires allowing each point to be recorded. Variations in temperature between thermocouple connections of the Al test plate of only $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$) were due to the high thermal conductivity of aluminum.

COLD WORKING AND ANNEALING

Each sheet was heated at $13.9^\circ\text{C}/\text{hour}$ ($25^\circ\text{F}/\text{hour}$) to 332°C (630°F), then held for 2 hours. Four to six sheets were annealed each batch, and the Al thermocouple sheet was placed on top to record the heat-up rate. During each batch annealing, the heat-up rate was $13.9^\circ\text{C}/\text{hr} \pm 0.5$ ($25^\circ\text{F}/\text{hr} \pm 1$). This was determined by the slope of the recorded temperature (Figure 17). Annealing treatments were followed by water quenching. Figure 18 represents the experimental test procedure flow diagram.

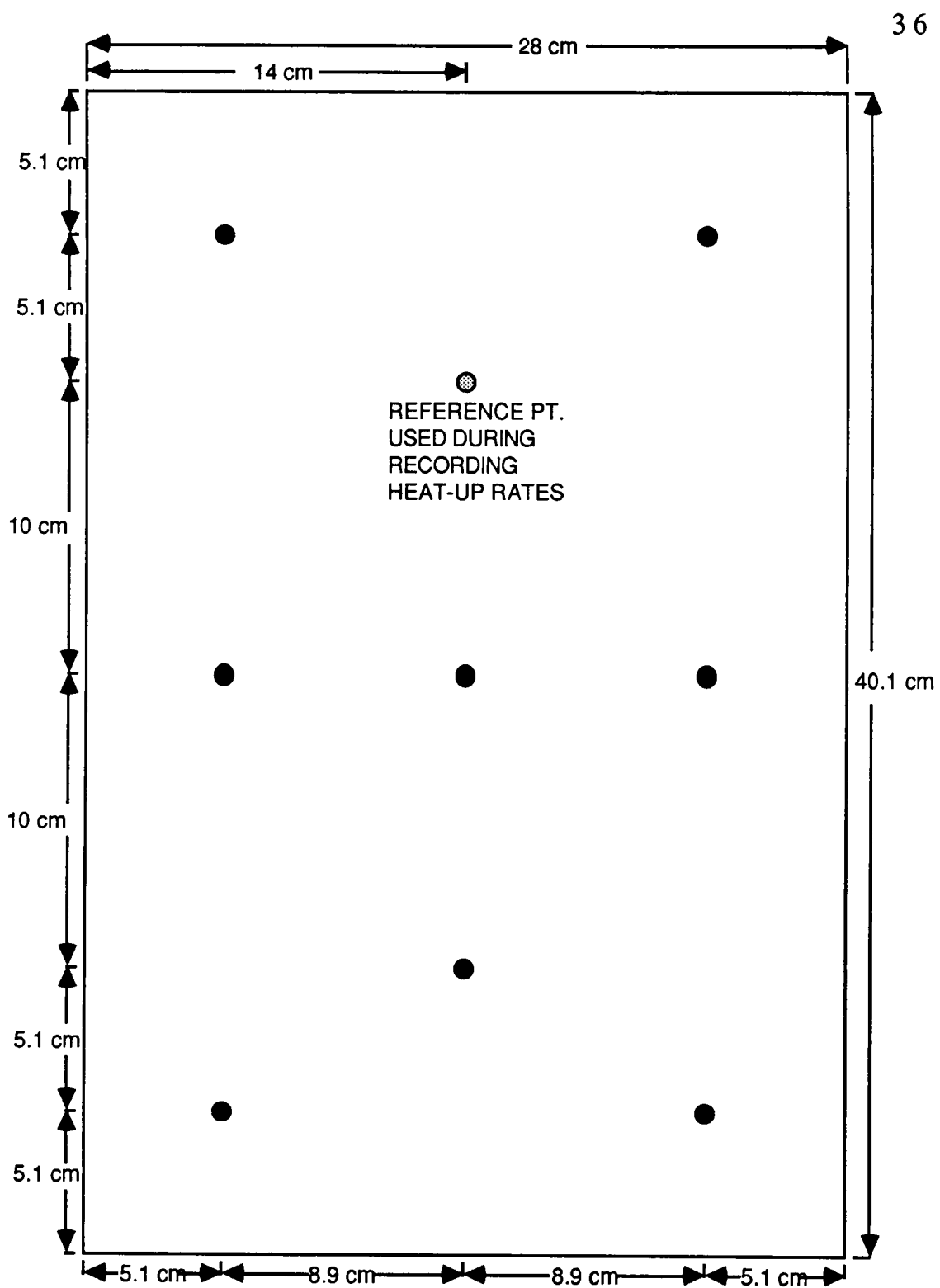


Figure 16. Thermocouple locations on aluminum test plate.

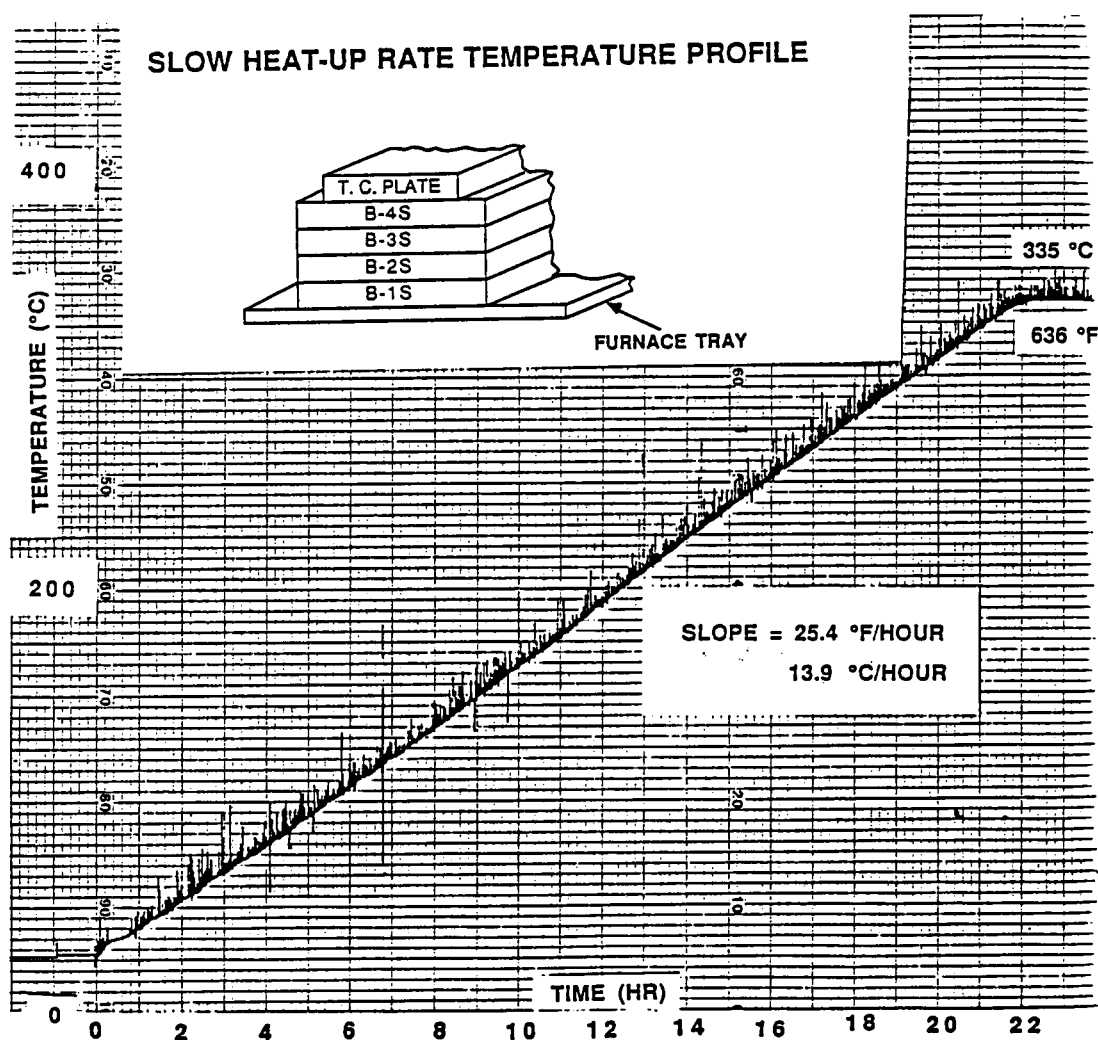


Figure 17. Time-temperature curve of slow heat-up annealing.

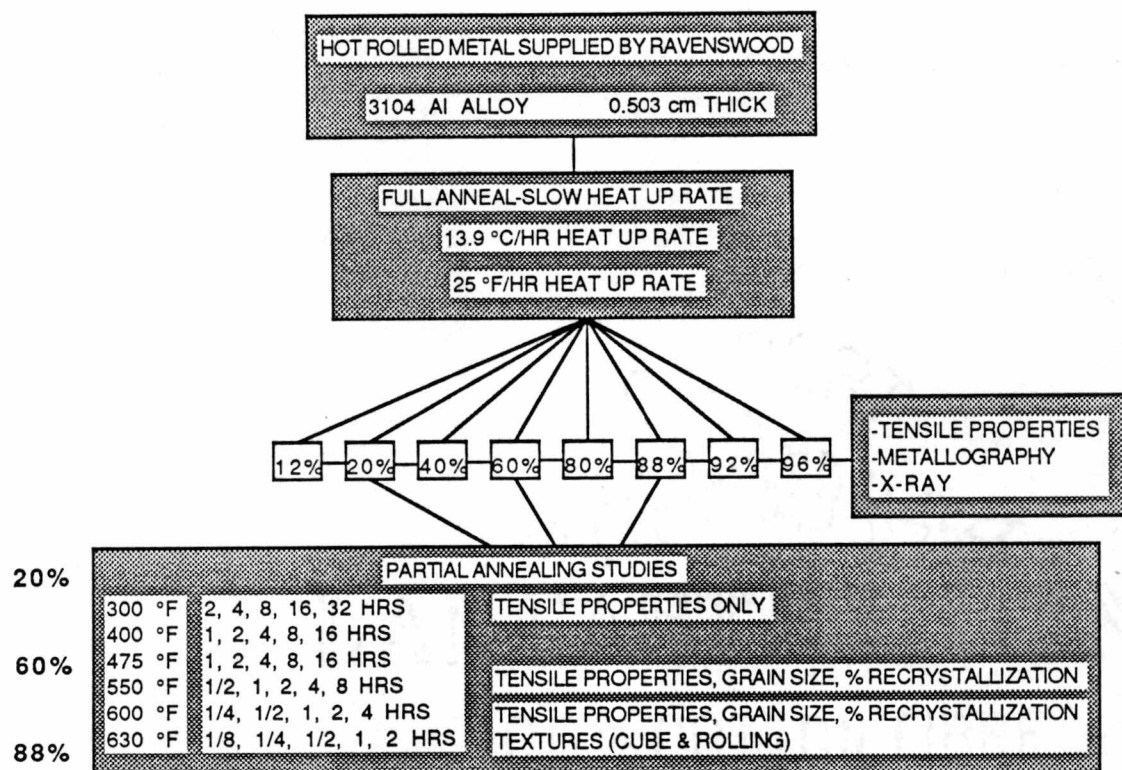


Figure 18. Flow diagram of experimental test procedure.

The annealed material was cold worked 12%, 20%, 40%, 60%, 80%, 88%, 92%, and 96% from the original thickness of 0.503 cm (0.198 in). Table 3 shows the thickness for each percent reduction. A variable speed rolling mill with 20.3 cm (8 in) diameter rolls was used to cold work the material. A micrometer was used to measure the thickness of both ends of the sheet after each pass. The maximum reduction per pass was 0.02 cm (0.008 in).

After cold working the material to approximately 50% reduction, the aluminum sheets began to flow easier during rolling. This could be due to some recovery taking place, although the sheets remained at about room temperature during the cold rolling process. A 24 hour time delay in cold rolling, without any adjustments in rollers, caused the material to become uncontrollable through buckling and curling. This was not completely understood,

Specimens for tensile tests were sheared to 1.9 cm x 25 cm (0.75 in x 10 in) at 0°, 45°, and 90° to the rolling direction as shown in Figure 19. Tensile specimens were sheared after each step. All tensile tests were conducted at Ravenswood Aluminum Corporation. Three specimens per condition were tested to check the repeatability of the measurements. Since the cold worked specimens showed good repeatability (yield strength ± 10 MPa (± 1.5 ksi) and elongation ± 2 %), only one specimen per condition was tested in the annealing studies.

After cold working, the materials for 20%, 60%, and 88% were annealed in a salt bath. A low temperature salt by Holden, tempering 312, was used. The thermocouple used to record the salt bath temperature was positioned 7.6 cm (3 in) from the top. Figure 20 shows the salt bath vertical

Table 3. Final thickness for percent reduction in cold working.

% Cold work *	** Calculated Final Thickness	
	cm	inch
12%	0.442	0.174
20%	0.401	0.158
40%	0.302	0.119
60%	0.201	0.079
80%	0.102	0.040
88%	0.061	0.024
92%	0.041	0.016
96%	0.020	0.008

* Hot rolled condition thickness = 0.503 cm (0.198 in).

** Final actual cold rolled thickness within ± 0.007 cm (± 0.003 in) of calculated thickness.

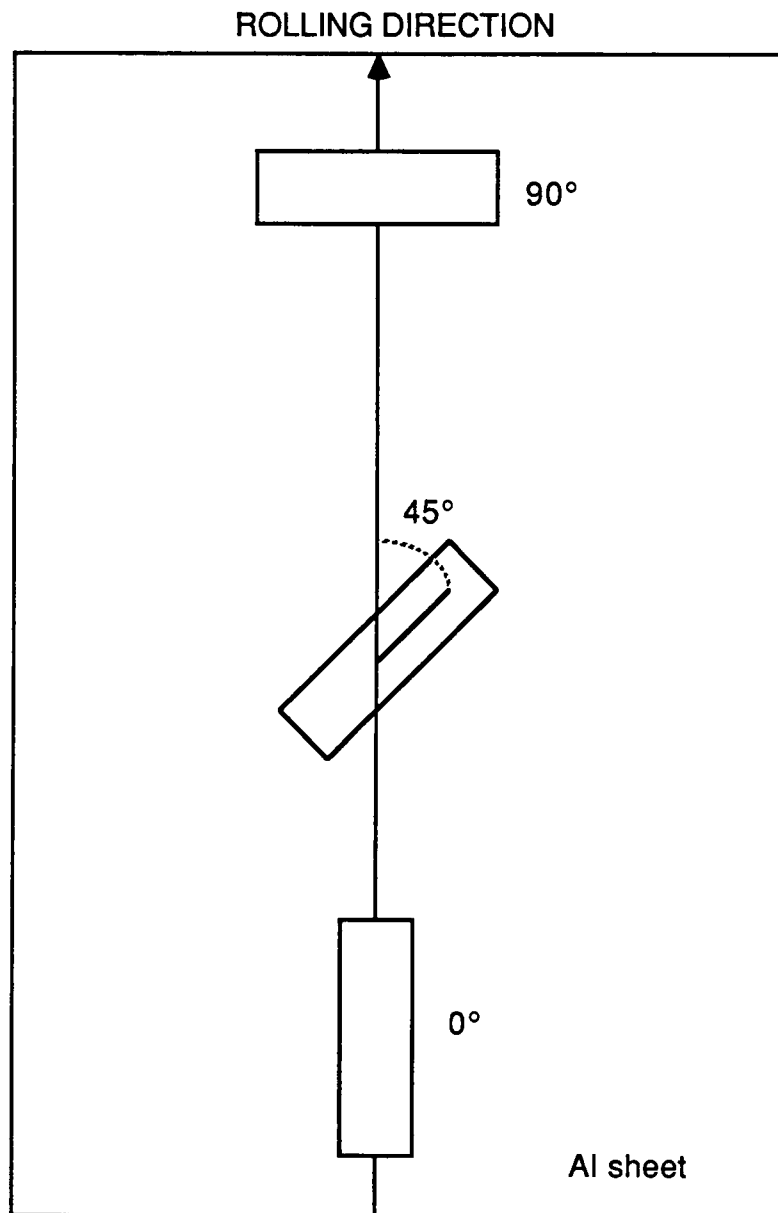


Figure 19. Location of tensile specimens.

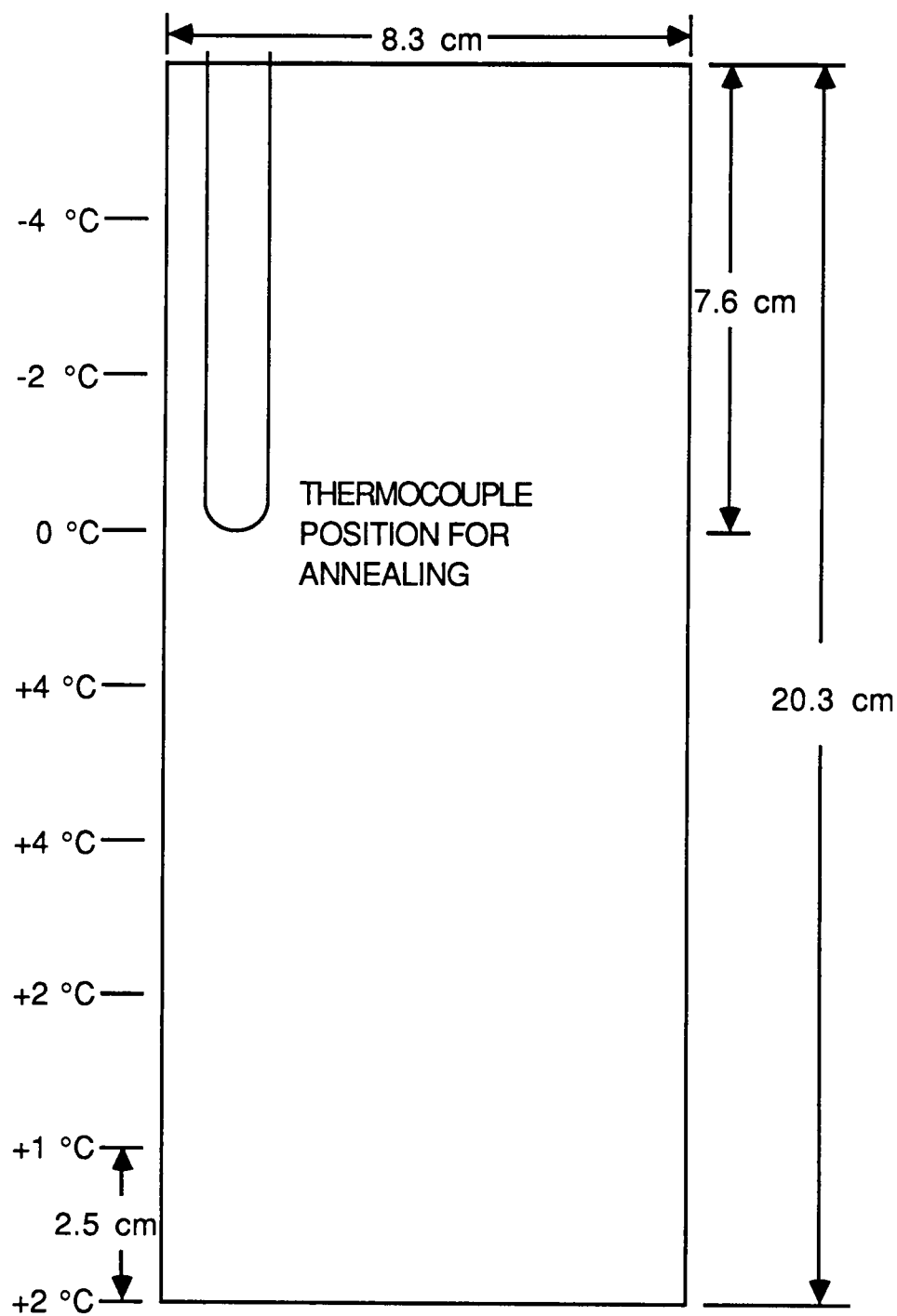


Figure 20. Vertical temperature difference of salt bath.

temperature profile. A K-type thermocouple in a stainless steel sleeve was connected to a chart recorder, and the temperature of the bath was monitored. A fire brick was used to cover the salt bath and the temperature remained within $\pm 2^{\circ}\text{C}$ ($\pm 4^{\circ}\text{F}$).

The tensile specimens (1.9 cm x 20 cm (0.75 in x 8 in)) were sheared from the cold worked material at 0° , 45° , and 90° to the rolling direction prior to salt bath annealing (Figure 19). A hole was drilled through one end of each tensile specimen. The tensile specimens (0° , 45° , and 90°) were connected together by copper wire with brass washers as separators. The specimens were annealed at 149°C (300°F), 204°C (400°F), 246°C (475°F), 288°C (550°F), 316°C (600°F), 332°C (630°F) from 1/8 hour to 32 hours, depending upon the annealing temperatures (Figure 18).

METALLOGRAPHY AND MICROSCOPY

For metallography, all samples were mounted in Bakelite. Three to five samples were placed in each mount depending upon sample size. Samples were cut by using a small cut-off wheel to 1.9 cm x 1.3 cm (0.75 in x 0.50 in) for mounting. The samples were taken from the tensile specimens at 90° to the rolling direction and mounted in the longitudinal direction (Figure 21). A hole was drilled through the center of each sample. A 2.54 cm (1 in) brass screw was placed through the hole of the samples using brass washers as separators. An opening to the back side of the mount was necessary for electrical connection during anodizing. Copper tubing (OD=0.48 cm) (3/16 in) of length approximately 1.9 cm (0.75 in) was used for his purpose. One end of the tubing was crimped using a vise which would

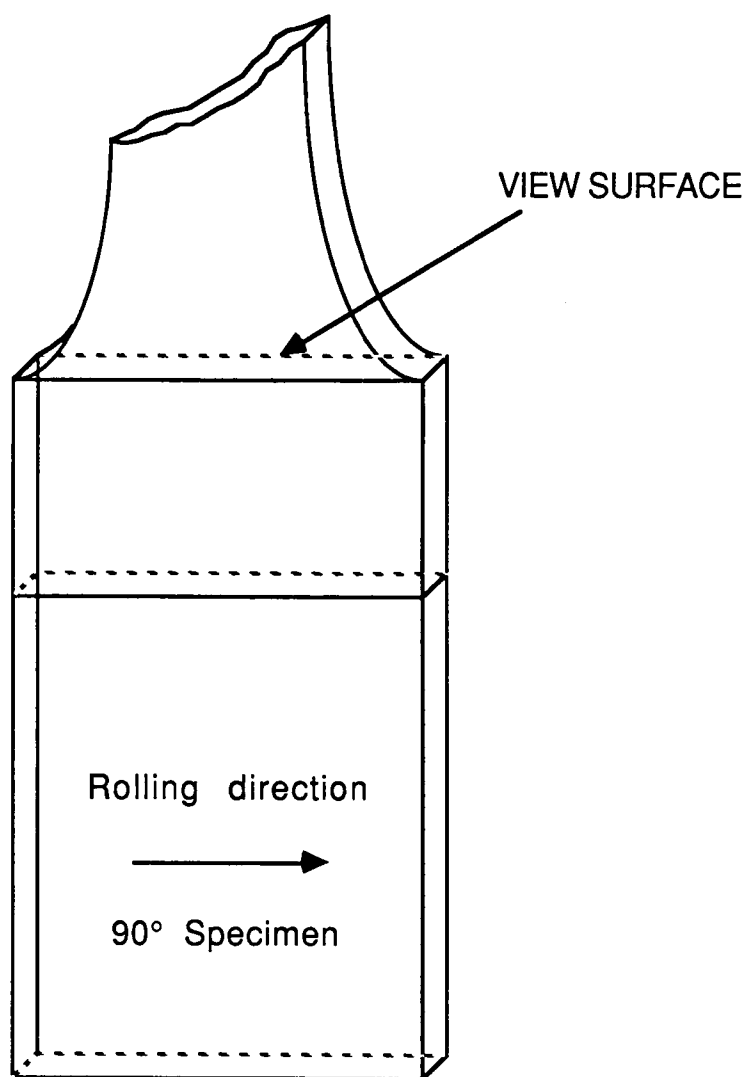


Figure 21. Location of metallographic samples in tensile specimens.

allow a hole to be drilled. This was positioned approximately in the center of screw assembly containing the samples. A nut was used to tighten the assembly to assure electrical contact between each sample. A carbon rod 0.318 cm (0.125 in) diameter was used to fill the copper tubing during mounting. A cross-section of the mounted assembly is shown in Figure 22. After mounting in Bakelite the back side was ground till the copper tubing could be seen or the mount was 1.6 cm (0.62 in) high. The carbon was drilled out and a thread was tapped. A screw in back of the metallographic mount was used for connection during anodizing.

The metallographic samples were ground using 1, 1/0, 2/0, and 3/0 emery papers and 600 grit SiC paper. The rough polishing was conducted using 1.0 μ alumina in distilled water. Magnesium oxide (MgO) powder with distilled water on a microcloth was used as the final polishing step to produce a surface finish which could be anodized. Without this, the samples could not be anodized.

For 3000 series aluminum alloys, an anodizing technique is used for revealing grain structure. During anodizing a thin film of aluminum oxide is deposited on the polished surface. For this process to occur in an electrolytic cell, the sample is the anode and the cathode is an aluminum strip which was 1.91 cm (0.75 in) wide. A special holder made of PVC allowed immersion in the electrolyte (Figure 23). Barker's reagent, 1.8% solution of fluoboric acid (HBF₄) in distilled water, was used as the electrolyte. The distance between the sample and the cathode was approximately 0.95 cm (3/8 in) [43]. A Raytheon DC power supply Model QRD40-2 was used as the electrical source. Approximately 20-30 volts D.C. were applied for 60-90 seconds at

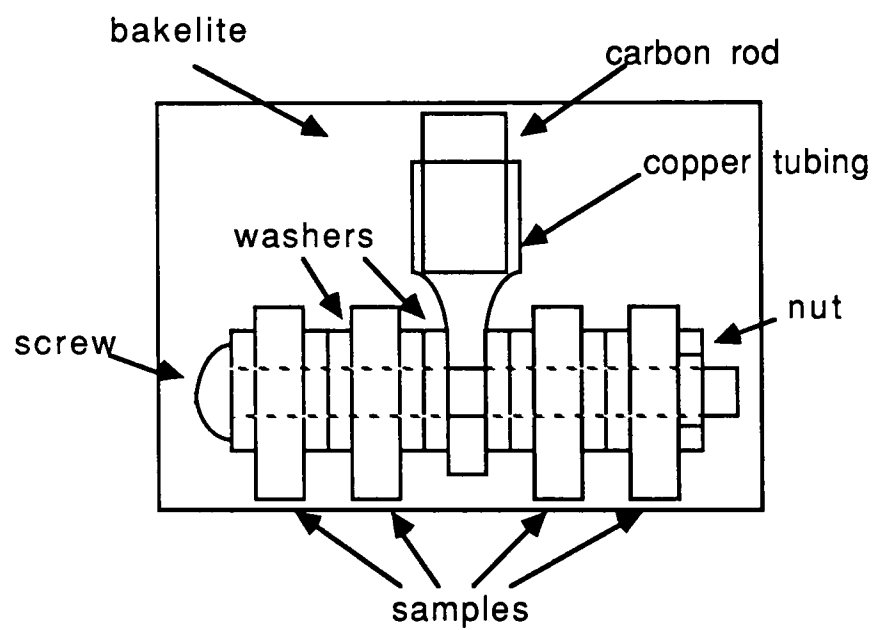


Figure 22. Assembly for mounting metallographic samples.

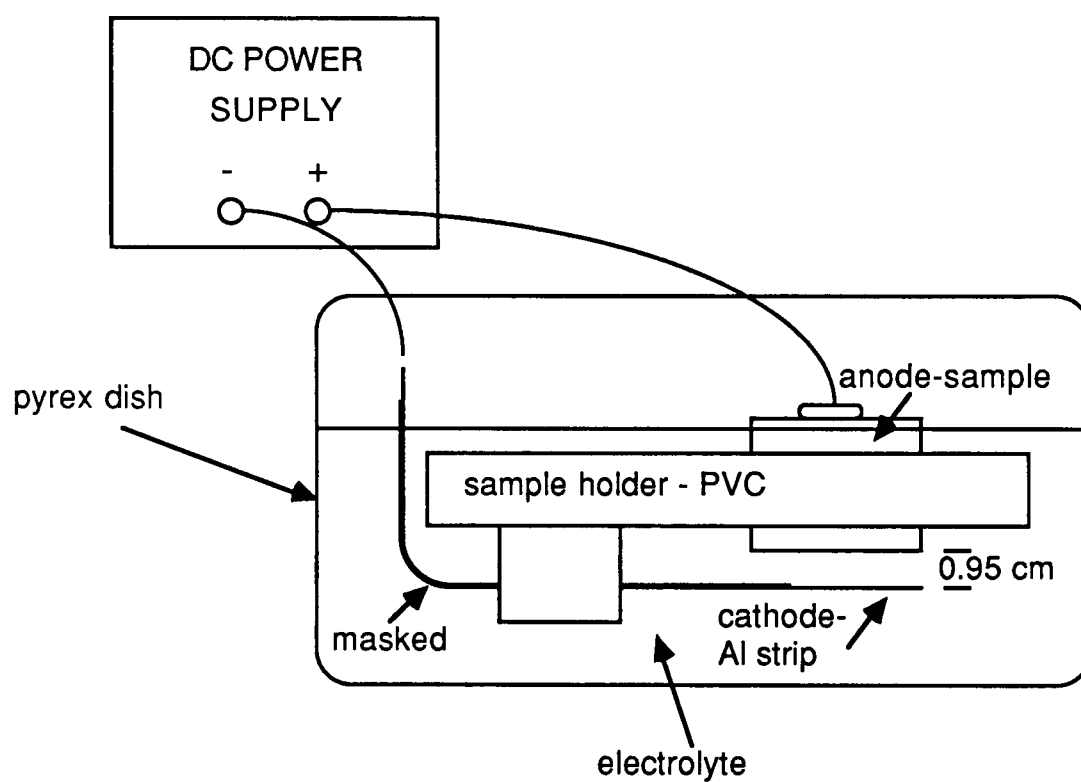


Figure 23. Schematic diagram of anodizing technique.

room temperature. Voltage and time used depended on the amount of sample area exposed. After anodizing was completed, the sample was rinsed in warm water and acetone then blown dry.

Samples of the rolling surface of the as-received (hot rolled) and slow anneal conditions were mounted and polished. The polished surfaces were examined initially unetched, then etched with 1% HF in distilled water to reveal inclusion distribution. A plane containing the longitudinal direction was examined to determine any variations in inclusion distribution with thickness (see Figure 21).

A Nikon Epiphot Optical microscope with polarized light was used to view the grain structure. Micrographs at 200x were taken for each condition for documentation and grain size measurements. Grain size measurements were taken only from the completely recrystallized samples obtained by annealing at 288°C (550°F), 316°C (600°F), 332°C (630°F) for 20%, 60%, and 88% cold work. The grain boundary intercepts were collected by using three concentric circles according to ASTM-E112 [44].

Fractured tensile samples were examined in a Cambridge S360 Scanning Electron Microscope (SEM) to determine the mode of failure. Inclusions in metallographic samples were also examined in the SEM using a Link AN-10000 energy dispersive x-ray analyzer.

X-RAY DIFFRACTION

Textures were determined using x-ray diffraction. A Rigaku Geiger-Flex diffractometer was used with Cu K α radiation and Ni filter. An x-ray tube voltage of 35 kV and a tube current of 20 mA were used for each sample.

The divergence beam slit of $1/2$ degree was used. The x-ray samples were taken from the grip section (i.e., not deformed in tensile test) of the 90° tensile specimens. The samples were cut to 4.76 cm x 1.9 cm (1.88 in x 0.75 in). Samples with thickness greater than 0.203 cm (0.080 in) were end-milled on one side to approximately 0.20 cm. Each sample was mounted on a glass slide, with the rolling direction horizontal, using double stick tape, and was carefully placed in the goniometer sample holder so that they were completely vertical and flush with the front of the holder. The area irradiated was the same for all samples.

The intensity of the {220} peak was also examined for the annealed tensile specimens and deep drawn cups. The high intensity of the {220} planes relates to the presence of deformation texture components. The scan angle of 2θ for the {220} peak was from 64.3 to 65.9 degrees.

A scintillation counter probe measured the intensity of the diffracted beam. The intensity measurements were recorded on chart paper and the integral of each peak was given by the computer. Using the step scan mode, the goniometer moved 0.01 degree per 5 sec dwell time. The scan angle of 2θ for the {200} peak was from 43.8 to 45.3 degrees. A high intensity {200} peak reflects a cube texture and low intensity is a non-cube texture. The {200} peak for each condition was compared against the {200} for a randomly oriented Al sample. A randomly oriented Al sample was prepared by mixing aluminum powder with Duco cement and pressing it flat into a holder. The x-ray intensity of this sample was compared with the intensity of a sintered aluminum sample. The intensities were approximately the same and the sintered sample was used to normalize the x-ray intensities.

Ravenswood Aluminum Corporation conducted cup drawing tests to determine the percent earing. The blanks for the annealing studies were heat treated in an air furnace. The percent earing was determined by the difference of the average high regions (ears) and low regions (troughs) divided by the average height the cup. The equations used are:

$$h_e \text{ (average ear height)} = \frac{h_{e1} + h_{e2} + h_{e3} + h_{e4}}{4} \quad (3.1a)$$

$$h_t \text{ (average trough height)} = \frac{h_{t1} + h_{t2} + h_{t3} + h_{t4}}{4} \quad (3.1b)$$

$$\text{Earing (\%)} = \frac{h_e - h_t}{\left[\frac{h_e + h_t}{2} \right]} \times 100 \quad (3.1)$$

The results from the cup drawing tests will be compared to the x-ray data to determine the type of earing.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter discusses the results obtained from the experimental work described in Chapter 3. It is divided into three sections: Mechanical Properties, Microstructures, and Texturing.

MECHANICAL PROPERTIES

The ultimate tensile strength (UTS), yield strength (YS), and elongation (% ductility) are the mechanical properties determined from the tensile tests. The data generated from the tensile tests at 0°, 45°, and 90° to rolling direction are given in tabular form in the Appendix.

The effect of the amount of deformation on the ultimate tensile strength is shown in Figure 24. At the hot rolled condition, the ultimate tensile strength is approximately 190 MPa (28 ksi). After annealing this value decreases to 165 MPa (24 ksi). It was shown microstructurally (next section) that the material is not completely recrystallized after hot rolling. Complete recrystallization does not generally occur after hot rolling at temperatures near the solidus temperature in 3XXX series alloys [16]. The ultimate tensile strength increases with increasing amount of cold working (reduction in thickness). After 96% cold work, it is approximately 385 MPa (56 ksi).

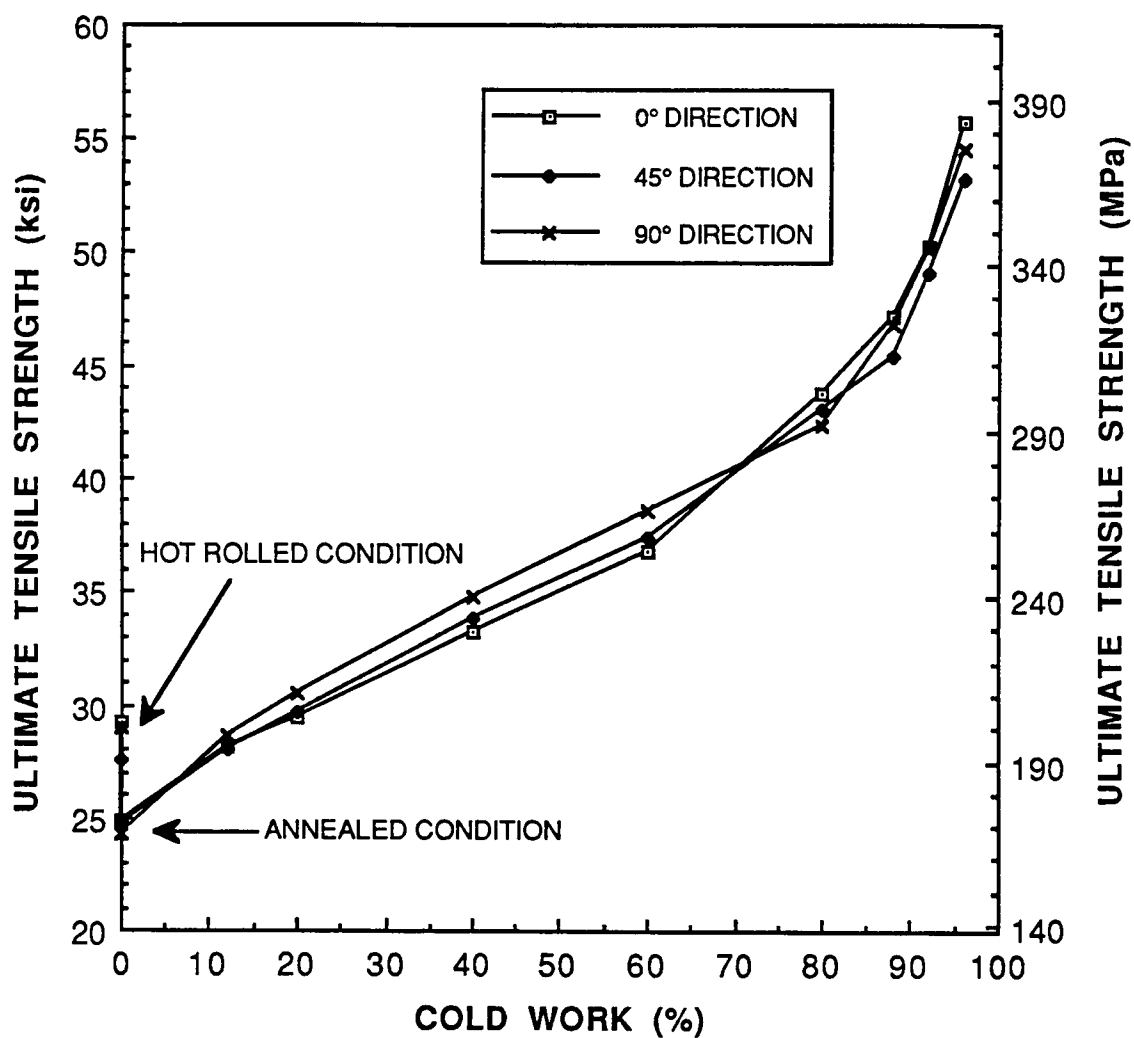


Figure 24. Ultimate tensile strength at 0°, 45°, and 90° to rolling direction vs. % cold work.

The effect of cold working on the yield strength is shown in Figure 25. It decreases from about 150 MPa (22 ksi) in the hot rolled condition to 88 MPa (12.7 ksi) in the annealed condition. After 96% cold work, the yield strength increases to 345 MPa (50 ksi). Figure 26 shows that the ductility, as represented by percent elongation, decreases with increasing cold work. It increases from about 20% after hot rolling to 26% after annealing. At 96% cold work, the ductility has decreased to approximately 2.5%.

The slope of the curves in Figures 24 and 25 are different in the first 10 percent reduction, in which the yield strength increases more rapidly than the tensile strength. At increasing plastic deformation, the yield strength approaches the tensile strength. This is very important in drawing operations. The stress generated by the load applied must be greater than the yield strength but below the tensile strength to avoid failure. If the difference between the tensile and yield strength is narrow, close control of the load applied during drawing is required.

Below 10% cold work, deformation is generally by slip only. Between 10% and 70%, the formation of deformation bands occurs [45]. At reductions greater than 70%, shear bands begin to form [45]. This change in mode of deformation could cause an increase in slope of the tensile and yield strength curves, as seen in Figures 24 and 25. Yi-Lin et al. suggested that the formation of deformation bands produces a stage where further hardening mechanisms become active [46]. The cause of this increase in mechanical properties is not completely clear and needs further investigation.

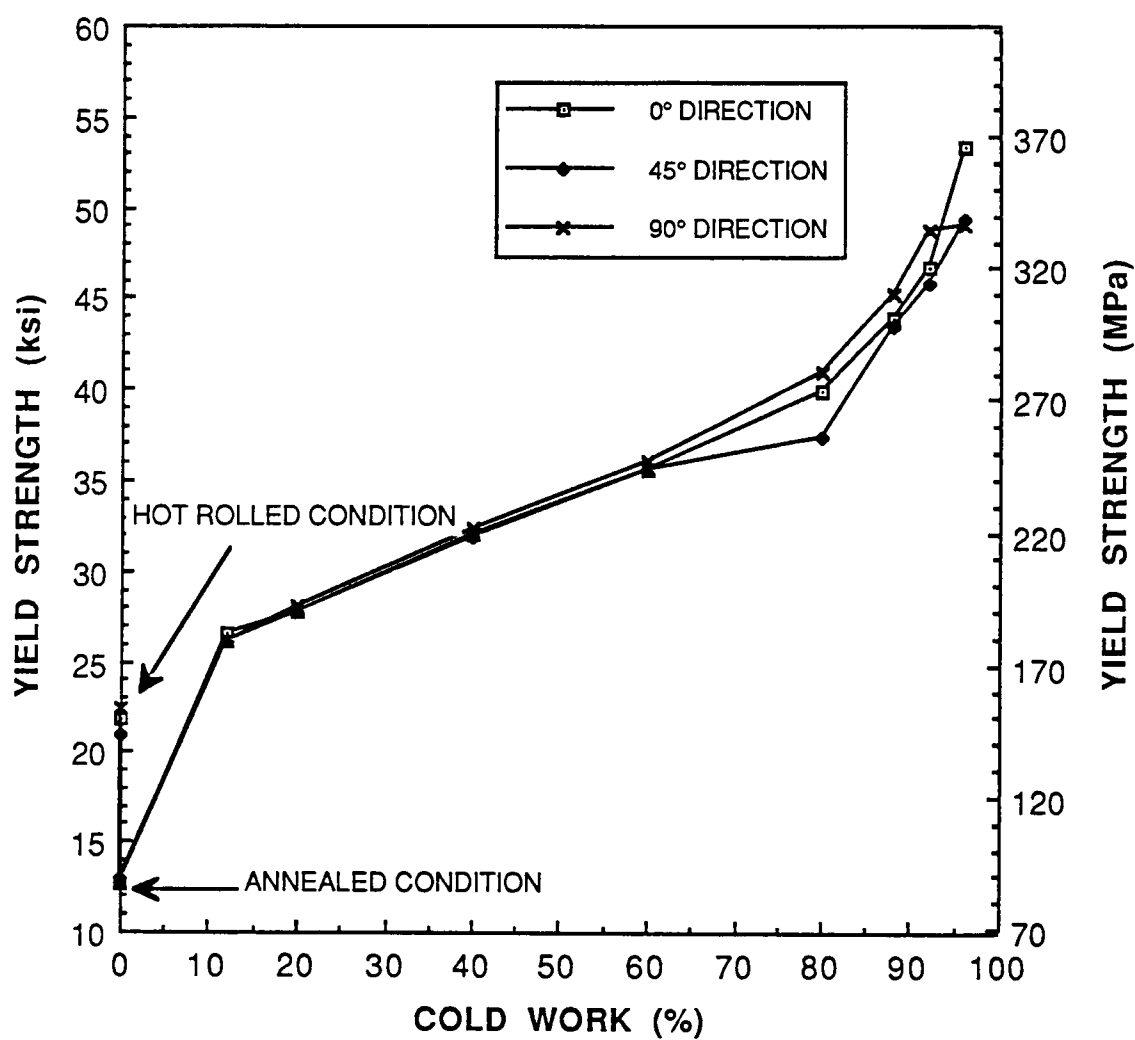


Figure 25. Yield strength at 0°, 45°, and 90° to rolling direction vs. % cold work.

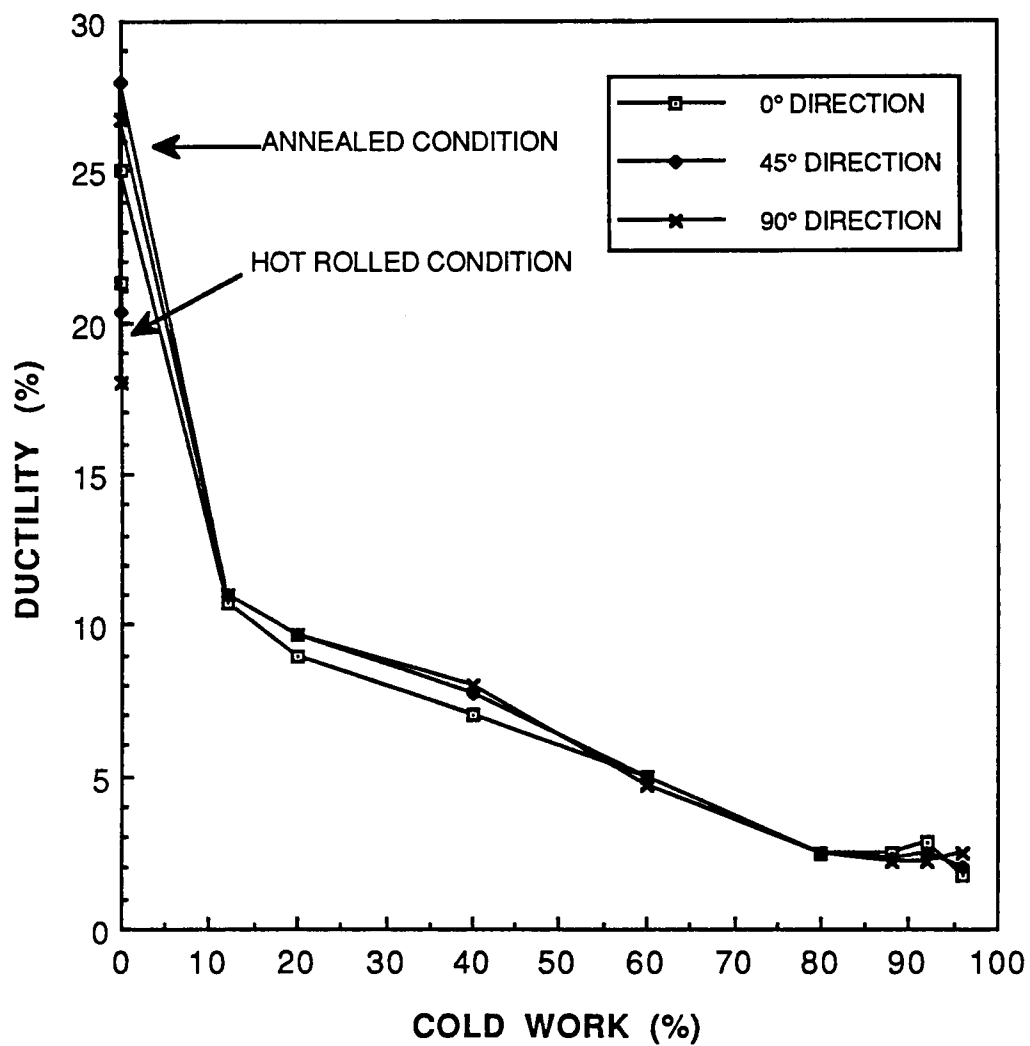


Figure 26. Ductility (%) at 0°, 45°, and 90° to rolling direction vs. % cold work.

Dover and Embury [47] reported that the ultimate tensile strength is 179 MPa (26 ksi), yield strength is 69 MPa (10 ksi), and elongation is 20% for 3004 aluminum alloy in the annealed condition. This is in close agreement with the results we obtained.

Examination of the data from the tensile tests shows very little variation in ultimate tensile strength, yield strength, and ductility with specimen orientation. This suggests that the material used in this research is essentially isotropic (planar) with respect to these mechanical properties.

Figure 27 represents a typical fracture surface of the tensile specimens. The fracture surface is dimpled due to fracture by microvoid coalescence. The nucleation of microvoids occurs at areas of high plastic deformation, which are associated with second phase particles. These were nucleated by both the large primary particles and the dispersoids. The size of the dimples is determined by number and distribution of microvoids that are nucleated.

Due to little change in mechanical properties with specimen orientation, only the properties of one direction were plotted for the annealing studies. Annealing studies were conducted for 20%, 60%, and 88% cold work.

At 20% cold work, the mechanical properties increased to 203 MPa (29.5 ksi) for ultimate tensile strength, 192 MPa (27.8 ksi) for yield strength, and the ductility has decreased to 9%. For 20% cold work, the mechanical properties as a function of annealing time at 149 °C (300 °F), 204 °C (600 °F), and 246 °C (475 °F) are shown in Figures 28-30. The recrystallization temperature for aluminum alloys is approximately 320 °C (600 °F) [21],

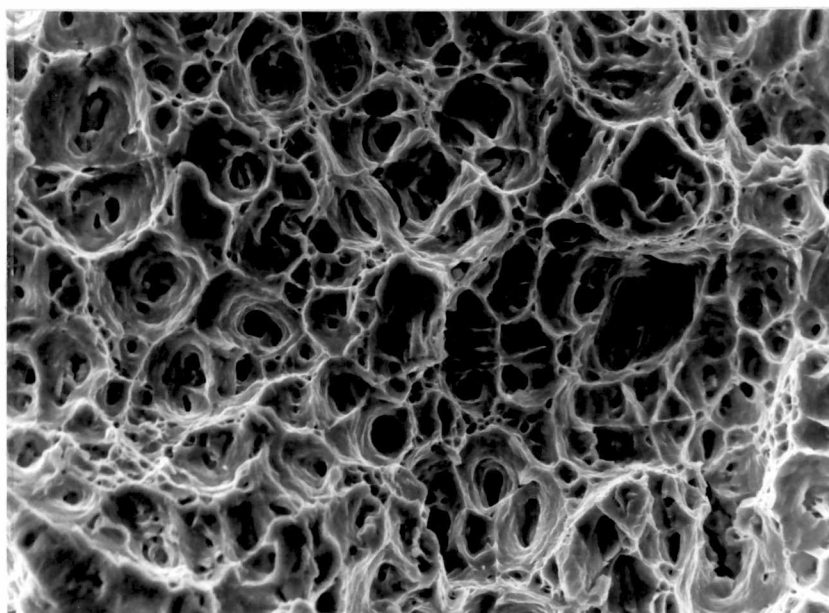
**SEM**50 μm

Figure 27. Dimple fracture surface (annealed condition) from tensile specimen. Rolling direction perpendicular to fracture surface. 550x.

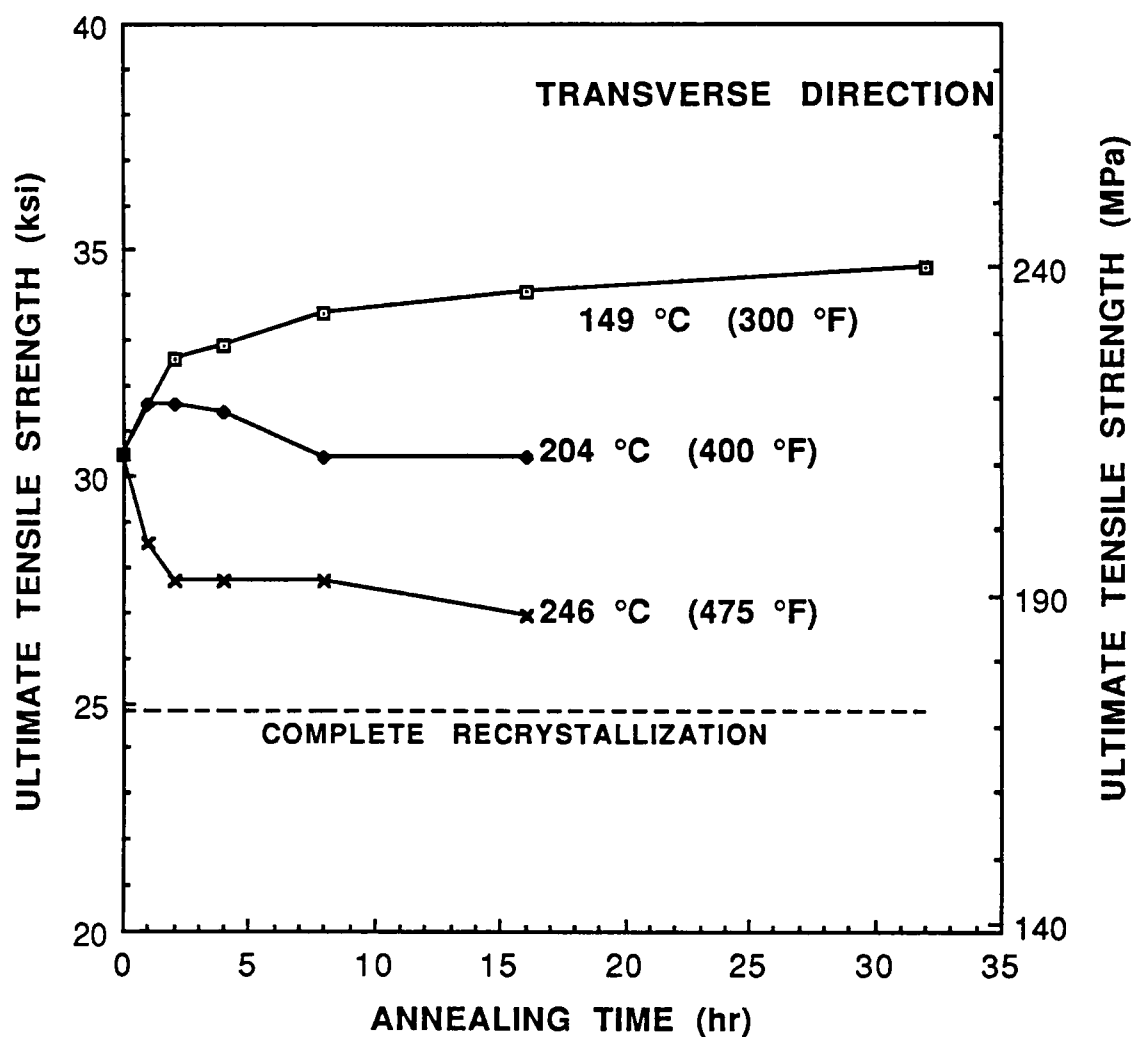


Figure 28. Ultimate tensile strength in the transverse direction for 20% cold work material at 149 °C, 204 °C, and 246 °C.

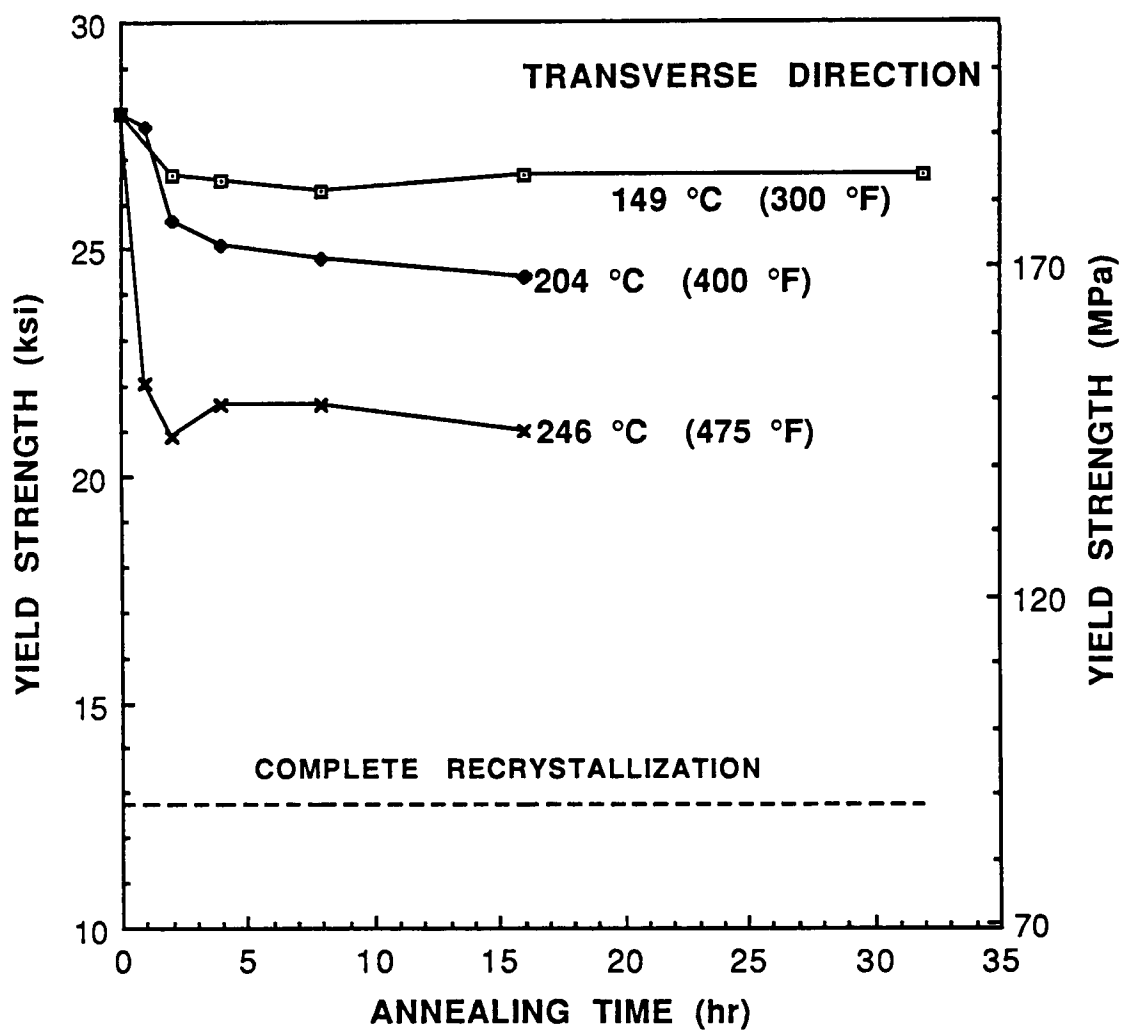


Figure 29. Yield strength in the transverse direction for 20% cold work material annealed at 149 °C, 204 °C, and 246 °C.

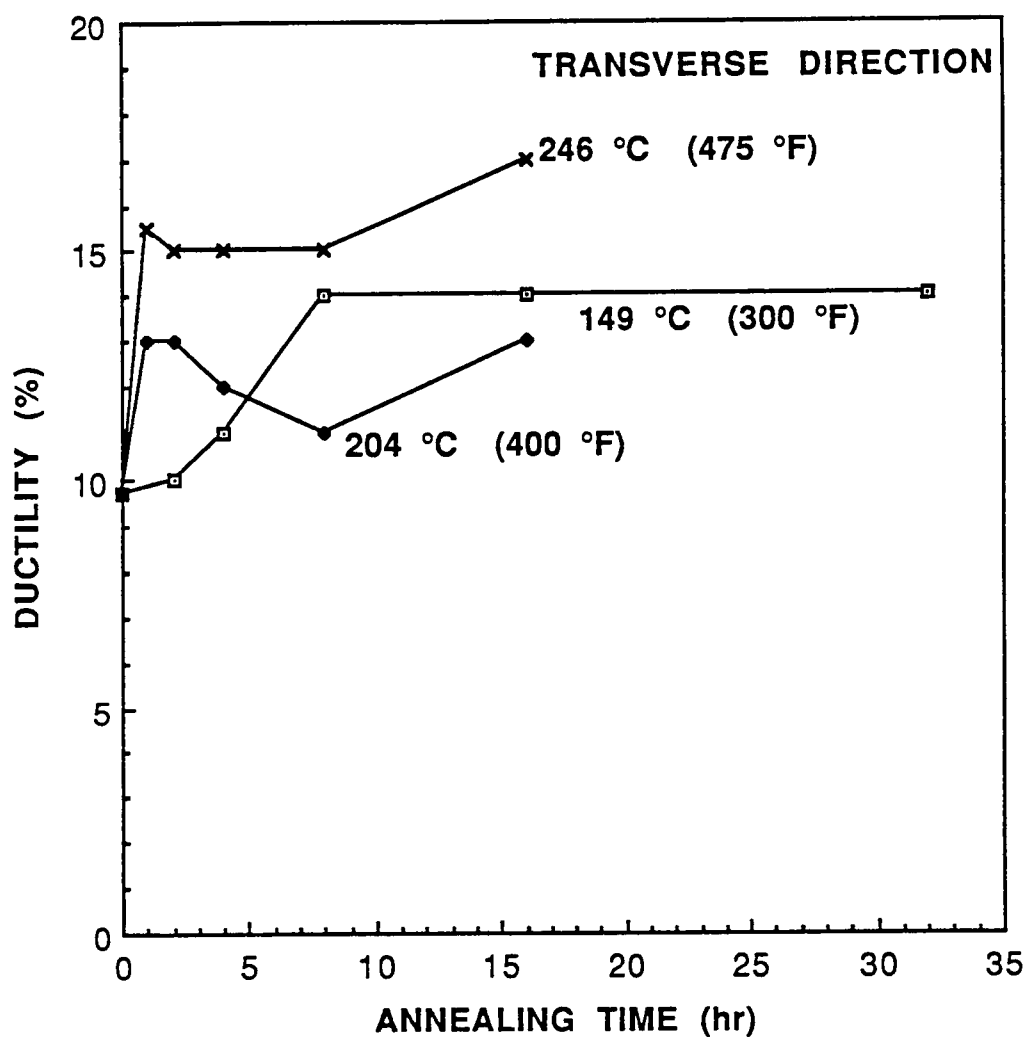


Figure 30. Ductility (%) in the transverse direction for 20% cold work material annealed at 149 °C, 204 °C, and 246 °C.

depending on prior deformation and alloy content. The three lowest annealing temperatures, 149 °C (300 °F), 204 °C (600 °F), and 246 °C (475 °F), are below this value. For 149 °C (300 °F), only a slight change in tensile and yield strength is observed, thus indicating only recovery has occurred after annealing 32 hours. Ductility increased slightly during recovery. For annealing at 204 °C (600 °F) and 246 °C (475 °F), recovery occurred with a slight decrease in tensile and yield strength properties after annealing 16 hours.

During recovery, the ultimate tensile and yield strength increases initially before the start of recrystallization. The magnitude of this change depends on the annealing temperature and times. The increase in ultimate tensile strength and the decrease in yield strength at 149 °C (300 °F) and 204 °C (400 °F) in Figures 28 and 29 are not completely understood.

The dashed line on the graphs indicates the mechanical properties after complete recrystallization. This was determined by microstructural examination and mechanical properties after slow annealing. In the annealed condition, the ultimate tensile strength is 171 MPa (24.8 ksi) and the yield strength is 88 MPa (12.7 ksi).

Figures 31-33 show the mechanical properties as a function of annealing time at 288 °C (550 °F), 316 °C (600 °F), and 332 °C (630 °F) for 20% cold work. At 288 °C (550 °F), the temperature is too low for complete recrystallization after annealing 8 hours (Figure 31). The final ultimate tensile strength is 185 MPa (26.8 ksi), and yield strength is 136 MPa (19.7 ksi). The ductility increased from 9% to 17%. An increase in temperature to 316 °C (600 °F) shows that complete recrystallization does not occur after 4 hours

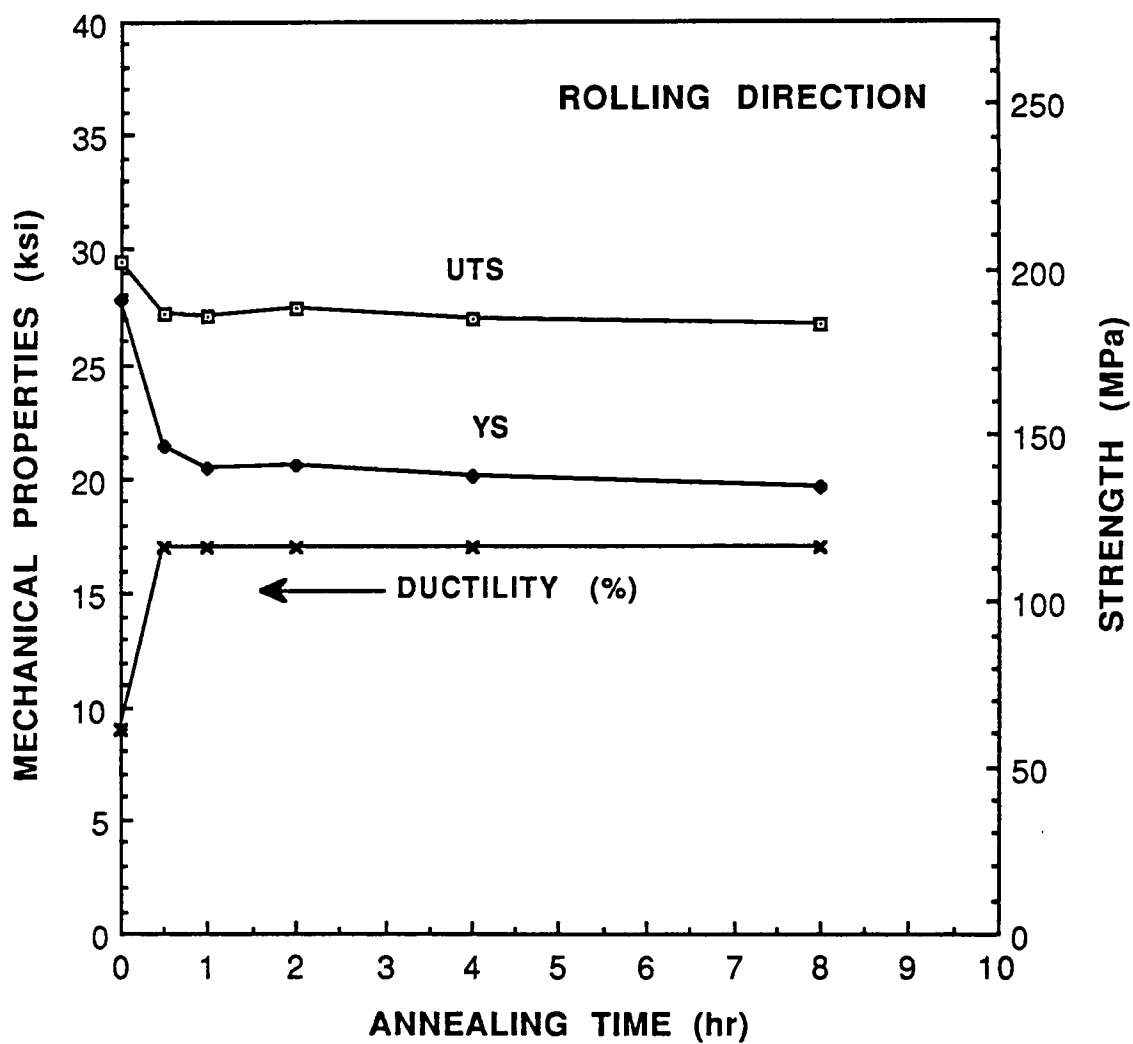


Figure 31. Mechanical properties in the rolling direction for 20% cold work material annealed at 288 °C (550 °F).

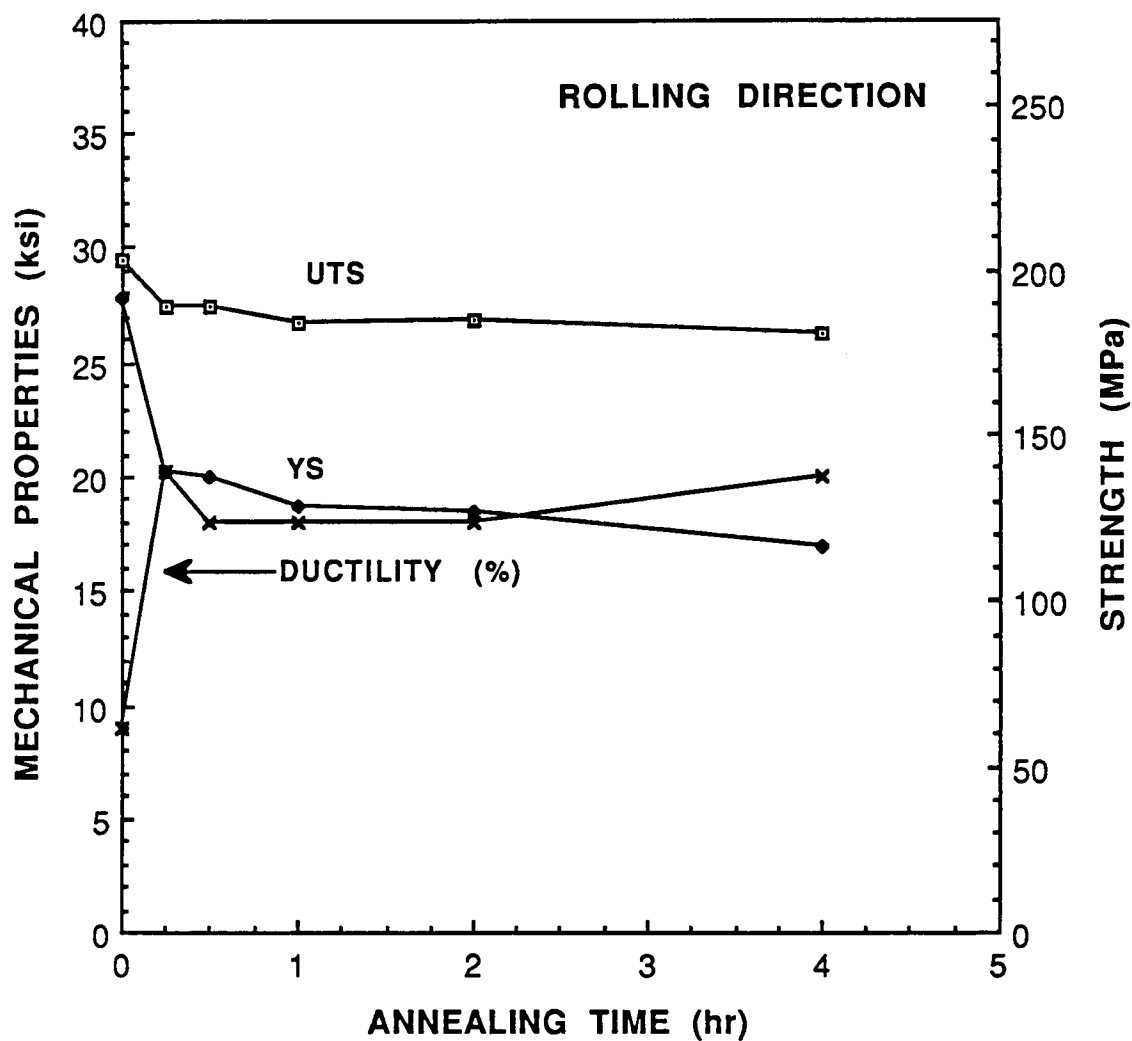


Figure 32. Mechanical properties in the rolling direction for 20% cold work material annealed at 316 °C (600 °F).

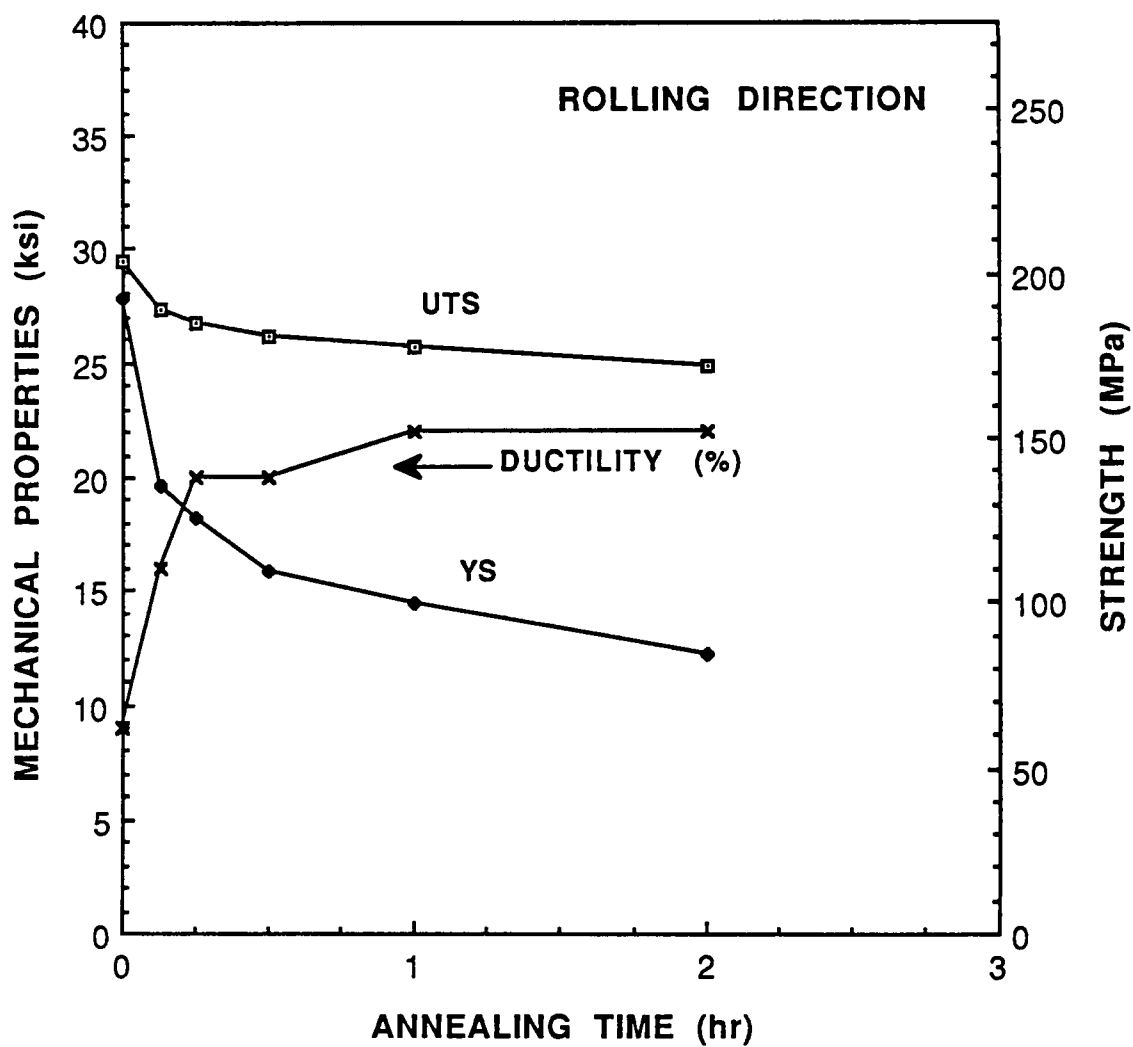


Figure 33. Mechanical properties in the rolling direction for 20% cold work material annealed at 332 °C (630 °F).

(Figure 32). The mechanical properties do not change appreciably by this increase in temperature. Only after annealing at 332 °C (630 °F) does complete recrystallization occur after 2 hours. The ultimate tensile strength is 172 MPa (24.9 ksi) and yield strength is 84 MPa (12.2 ksi) while the ductility increases to 22%. This is shown in Figure 33.

After 60% cold work, the ultimate tensile strength is 254 MPa (36.8 ksi), yield strength 245 MPa (35.6 ksi), and ductility is 4%. Figures 34-36 show the mechanical properties as a function of annealing time at the lower annealing temperatures for 60% cold work. At 149 °C (300 °F), 204 °C (600°F), and 246 °C (630 °F), the temperatures are below the recrystallization temperature and only recovery occurs after annealing. The curve for 204 °C (600°F) annealing temperature shows an increase in ultimate tensile strength and yield strength and a decrease in ductility at 8 hours. This is the only condition for which we observed this and it could be due to the precipitation of the α -dispersoid particles.

At the higher annealing temperatures, complete recrystallization occurred. Figure 37 shows the mechanical properties as a function of annealing time at 288 °C (550 °F). After 4 hours the material is completely recrystallized. Figure 38 shows the results after annealing at 316 °C (600 °F). Complete recrystallization occurs after 1/2 hour. After annealing at 332 °C (630 °F) complete recrystallization occurs after 1/8 hour (Figure 39). The final ultimate tensile strength is 172 MPa (25 ksi), yield strength 76 MPa (11 ksi) and the ductility has increased to 25%. Thus, an increase in the reduction in thickness decreased the temperature and time required for complete recrystallization.

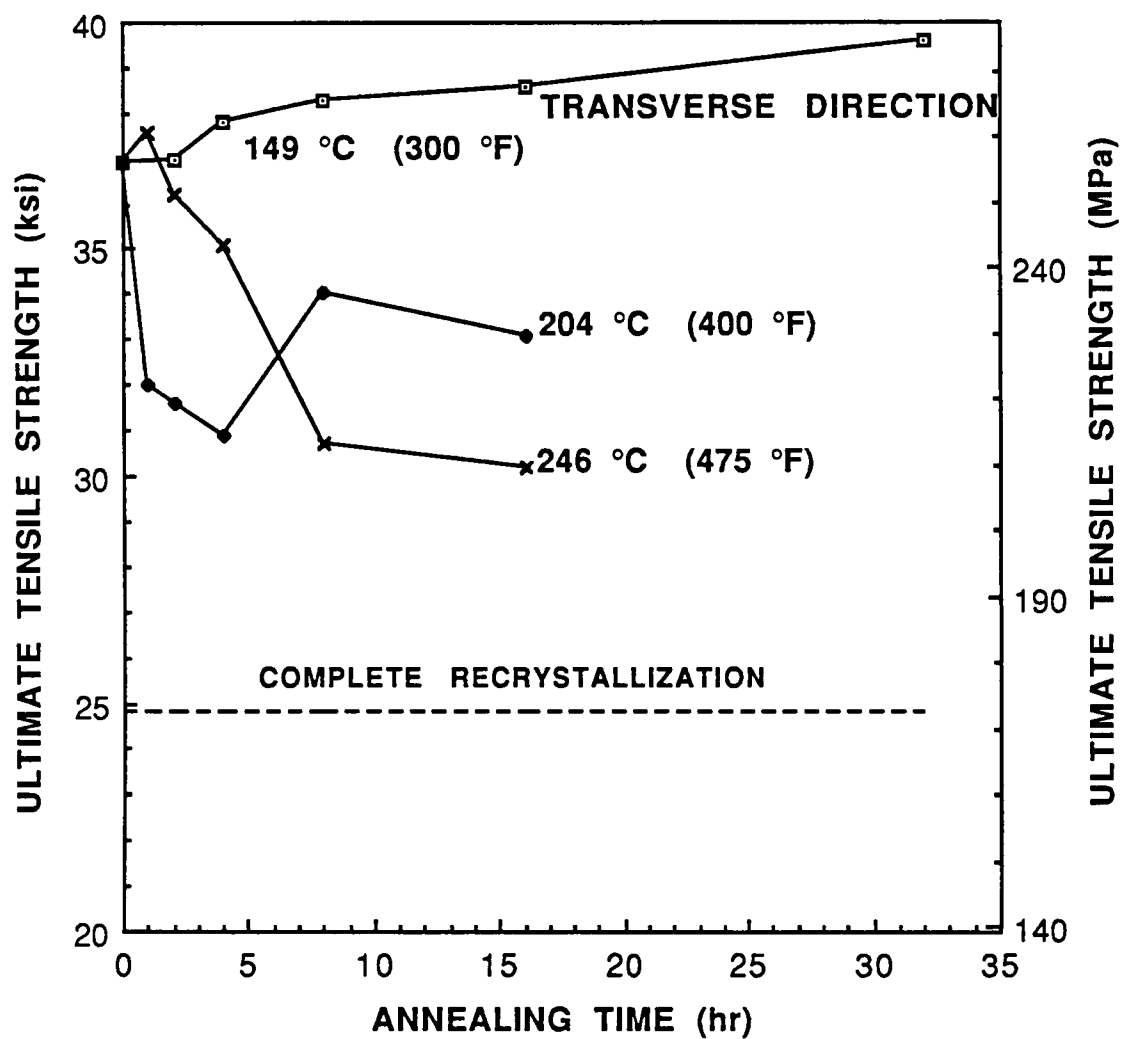


Figure 34. Ultimate tensile strength in the transverse direction for 60% cold work material at 149 °C, 204 °C, and 246 °C.

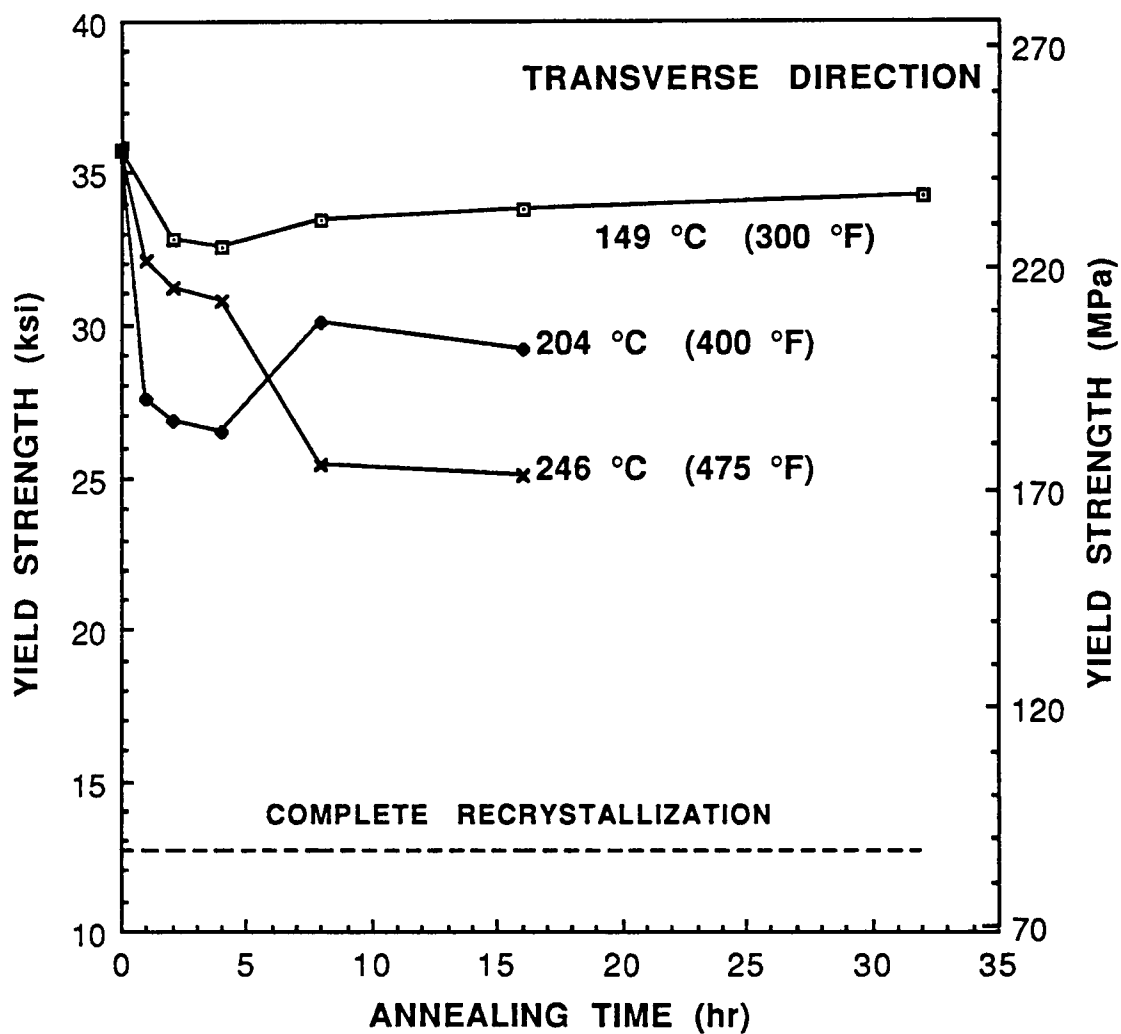


Figure 35. Yield strength in the transverse direction for 60% cold work material annealed at 149 °C, 204 °C, and 246 °C.

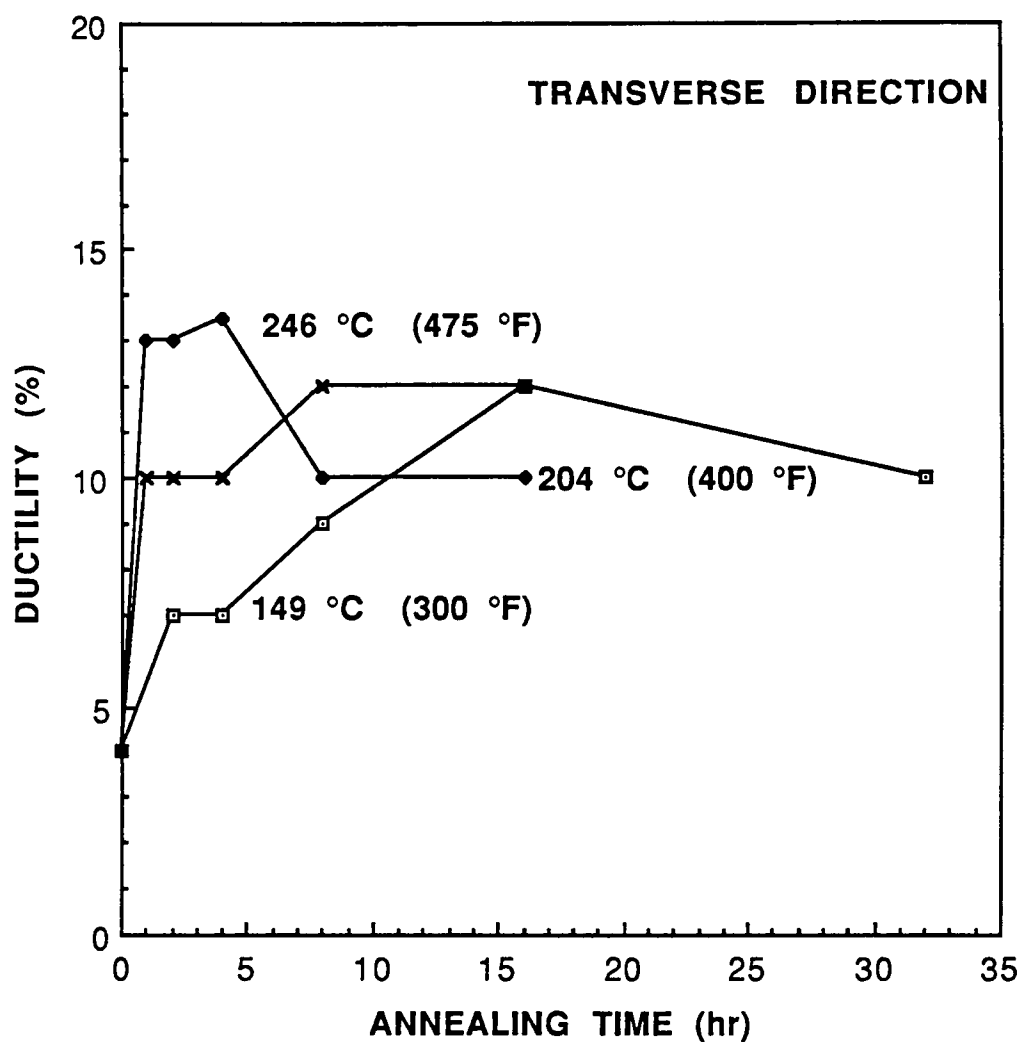


Figure 36. Ductility (%) in the transverse direction for 60% cold work material annealed at 149 °C, 204 °C, and 246 °C.

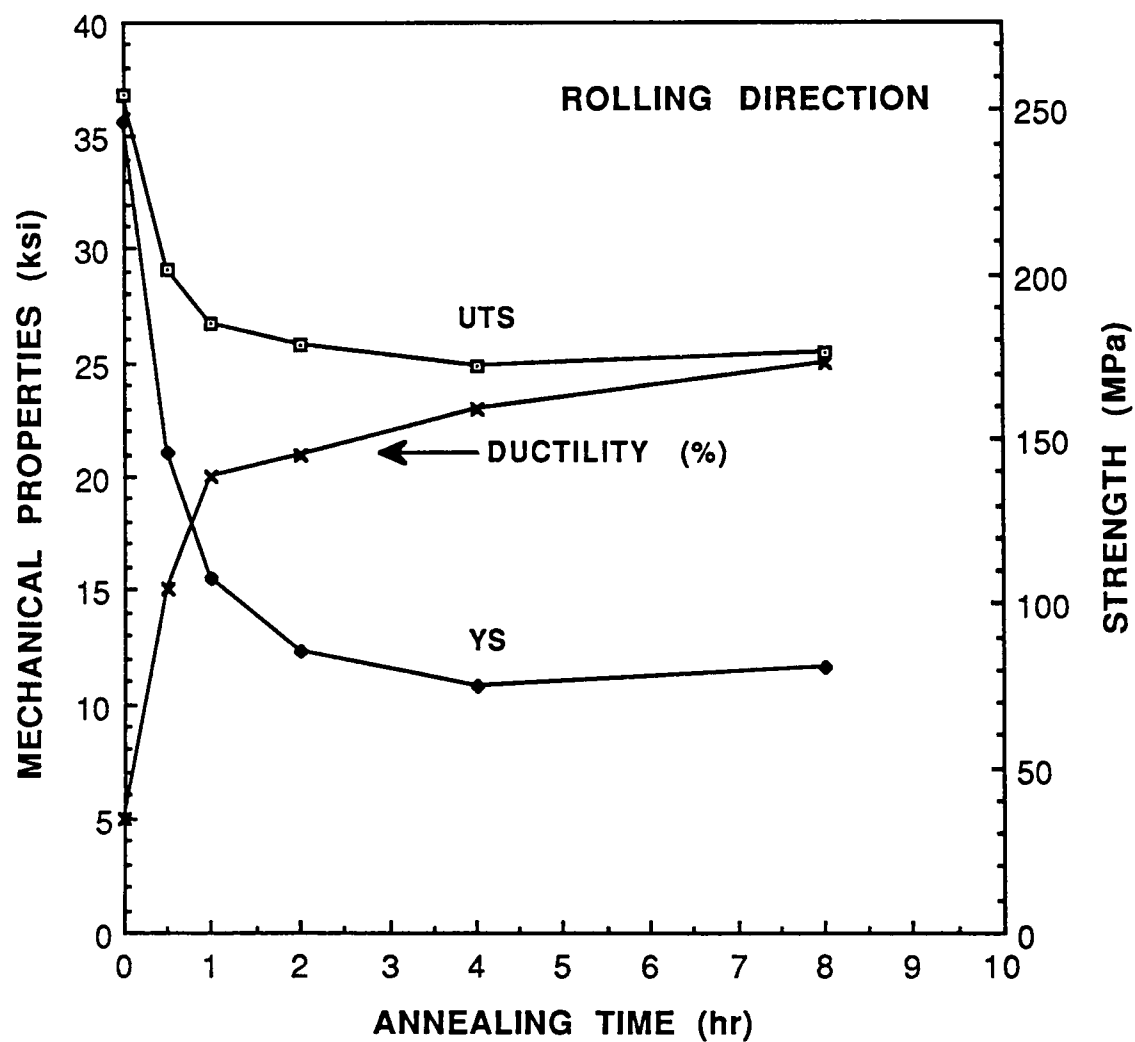


Figure 37. Mechanical properties in the rolling direction for 60% cold work material annealed at 288 °C (550 °F).

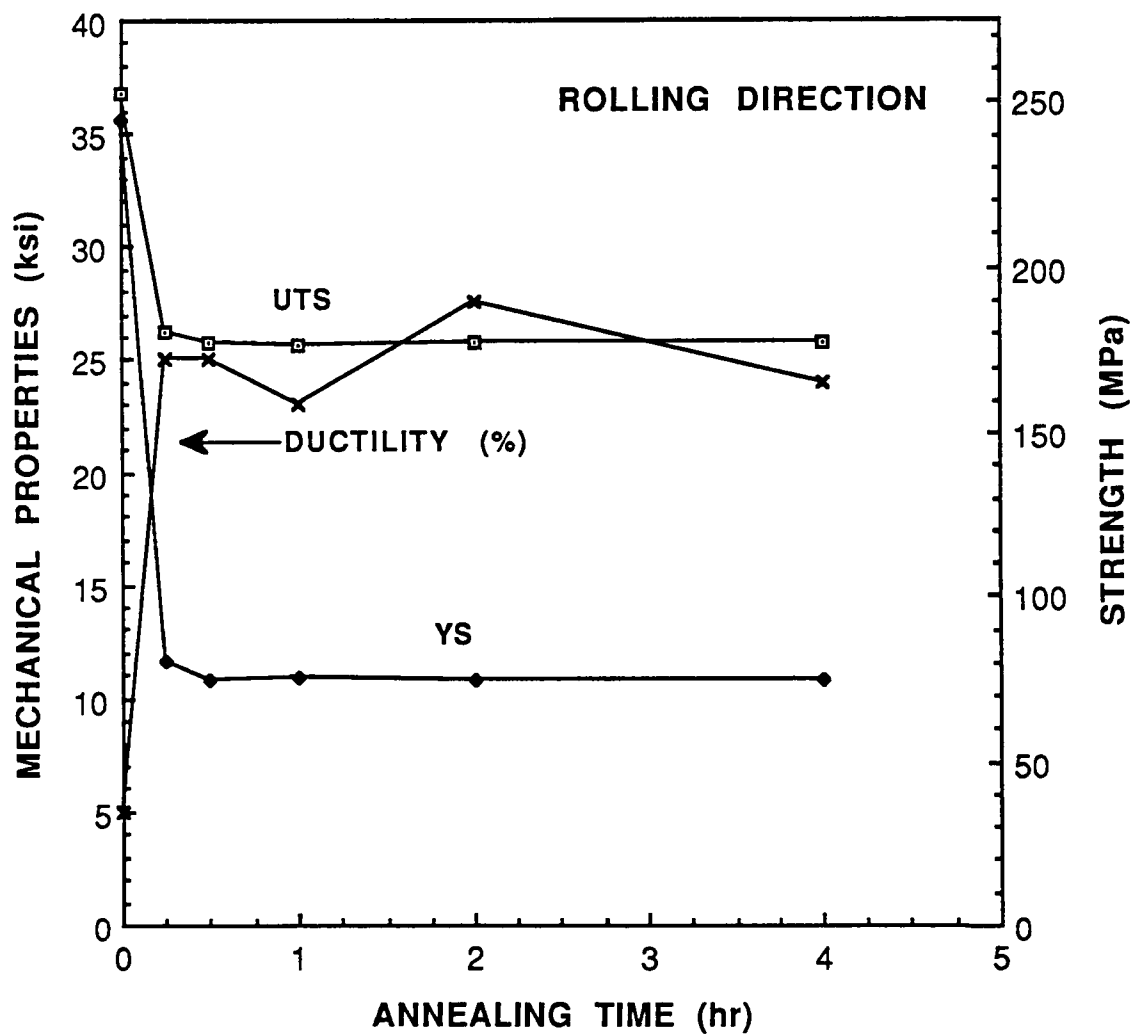


Figure 38. Mechanical properties in the rolling direction for 60% cold work material annealed at 316 °C (600 °F).

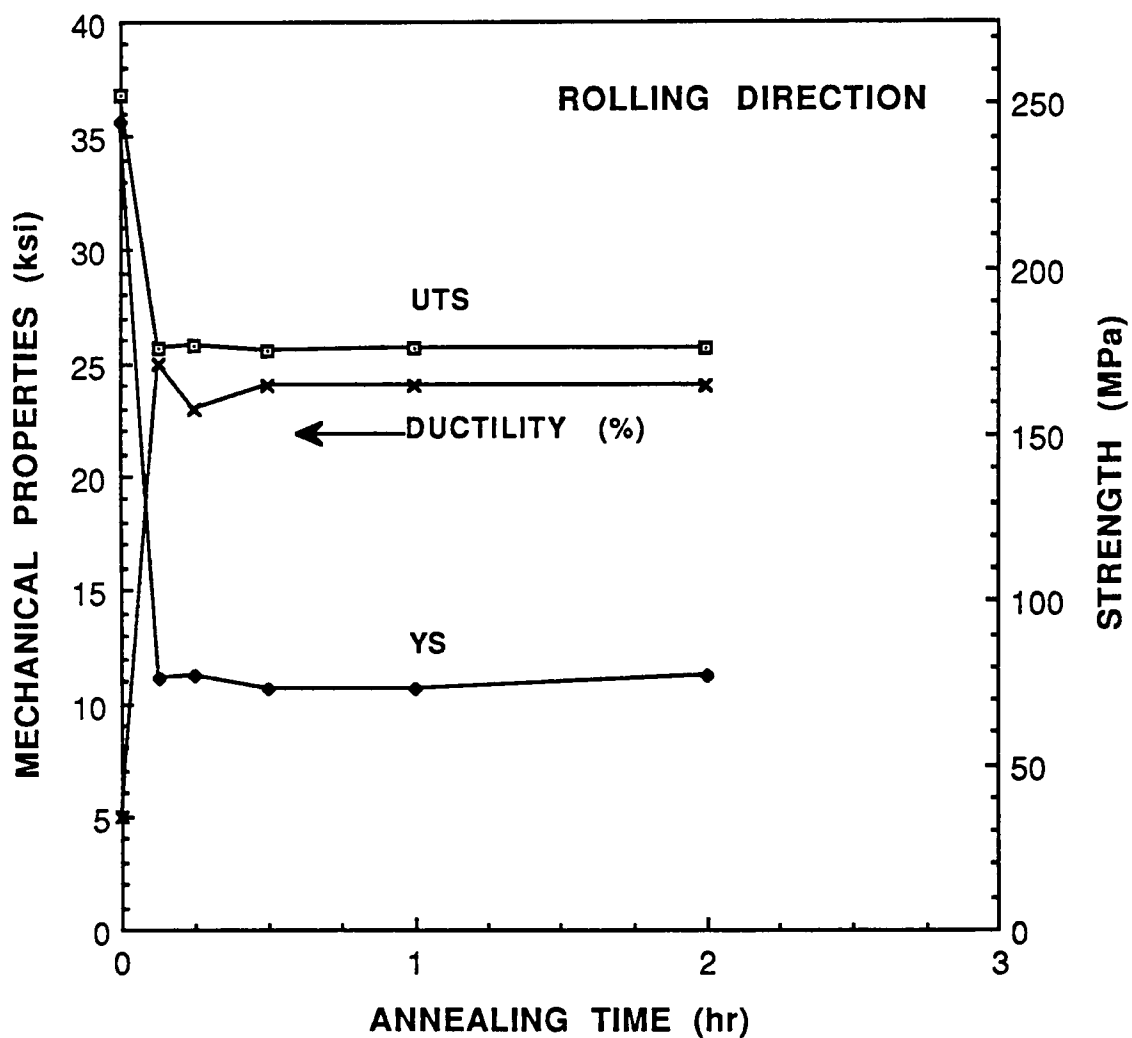


Figure 39. Mechanical properties in the rolling direction for 60% cold work material annealed at 332 °C (630 °F).

After cold working 88% reduction, the ultimate tensile strength increased to 325 MPa (47.2 ksi), the yield strength to 302 MPa (43.9 ksi), and the ductility decreased to 2.5%. The results of mechanical properties after annealing for 88% cold work are shown in Figures 40-45. The ultimate tensile strength, yield strength, and ductility at annealing temperatures of 149 °C (300 °F), 204 °C (600 °F), and 246 °C (475 °F) as a function of annealing time is shown in Figures 40-42, respectively. Only recovery occurs at these temperatures.

At the higher annealing temperature of 288 °C (550 °F), complete recrystallization occurs in 1 hour, as shown in Figure 43. Figures 44 and 45 show that recrystallization occurs within 1/4 hour at 316 °C (600 °F) and 1/8 hour at 332 °C (630 °F). The ultimate tensile strength has decreased to 177 MPa (25.5 ksi), yield strength to 83 MPa (12.1 ksi), and the ductility has increased to 20%.

MICROSTRUCTURES

The microstructures were examined by optical and scanning electron microscopy for each condition. Complete recrystallization was determined through evaluation of microstructures and mechanical properties.

The particle distribution was examined for the hot rolled and slow anneal conditions. The surface just below the rolling plane is shown in Figure 46 in the as-polished condition. Very little difference in particle size and distribution was observed between the two conditions. Energy dispersive x-ray analysis showed that the large particles were AlMn₆ and the α -dispersoid phase containing silicon. The α -phase was either partially or

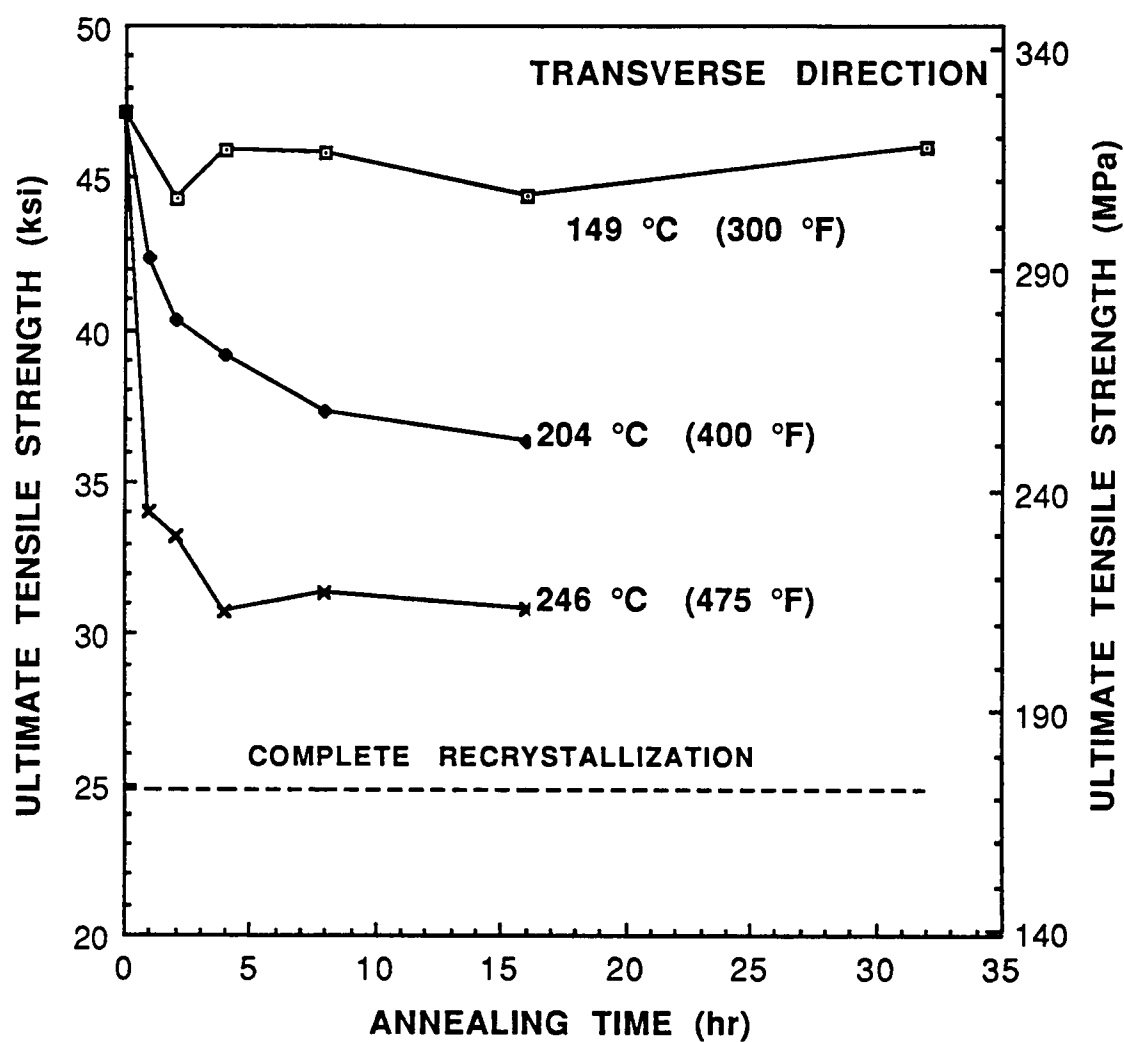


Figure 40. Ultimate tensile strength in the transverse direction for 88% cold work material annealed at 149 °C, 204 °C, and 246 °C.

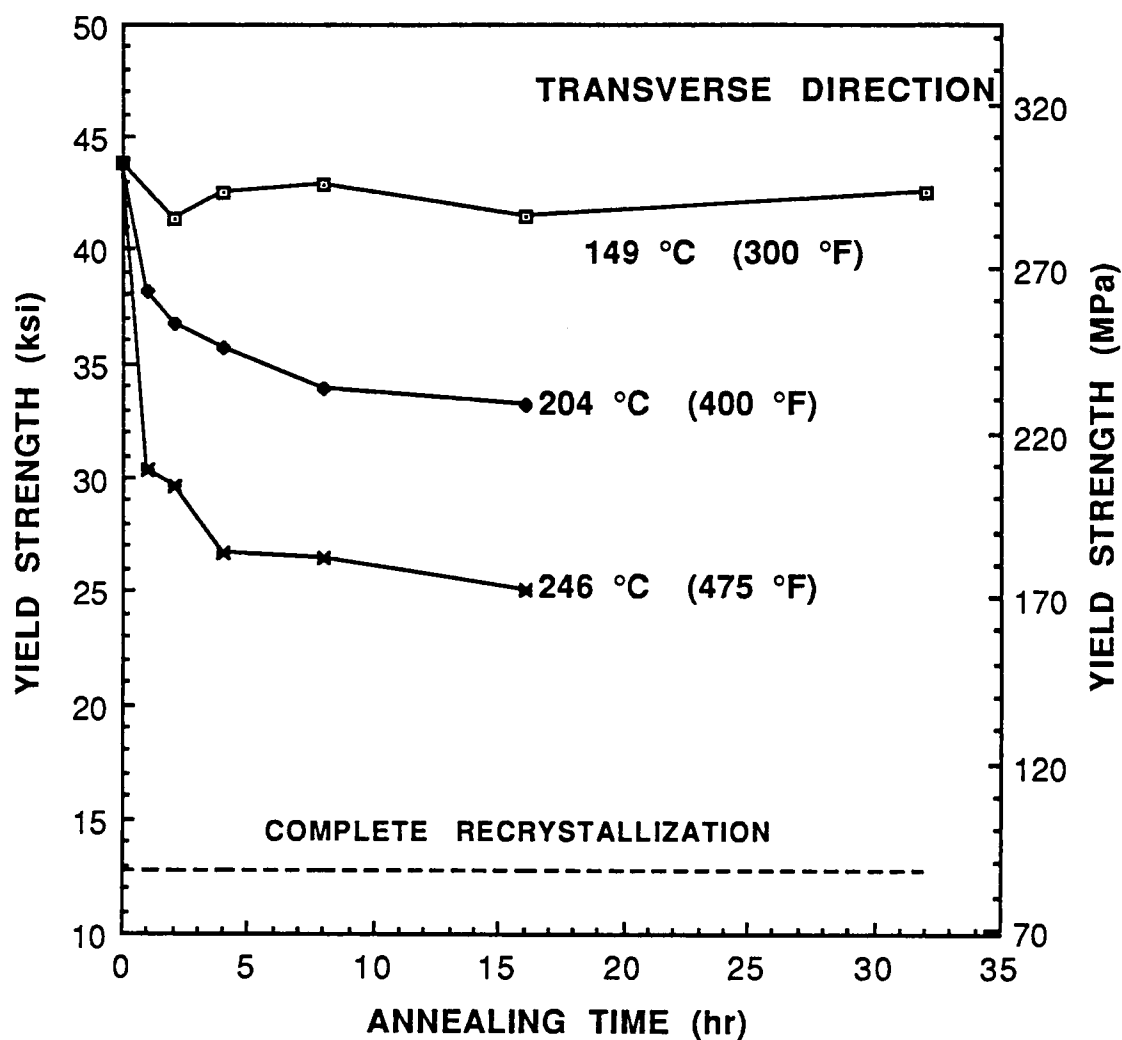


Figure 41. Yield strength in the transverse direction for 88% cold work material annealed at 149 °C, 204 °C, and 246 °C.

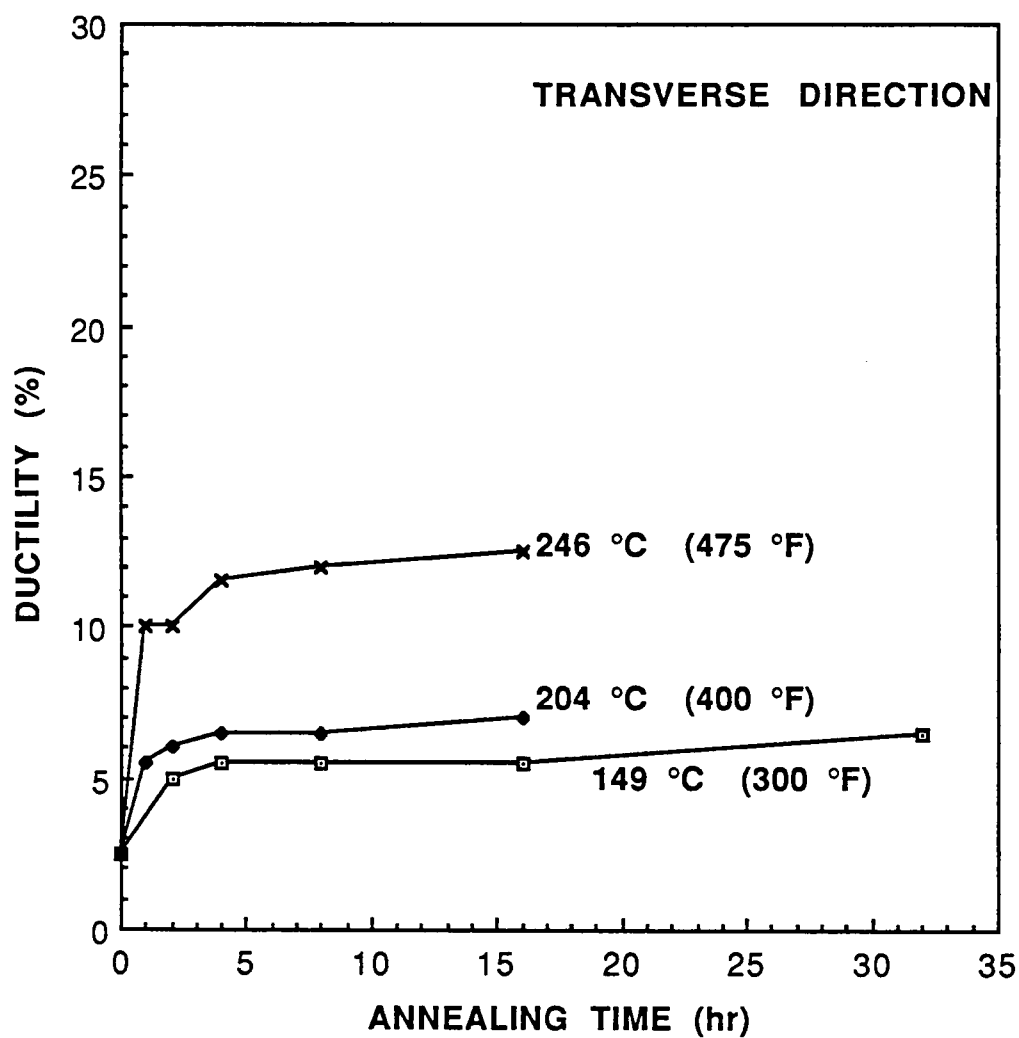


Figure 42. Ductility (%) in the transverse direction for 88% cold work material annealed at 149 °C, 204 °C, and 246 °C.

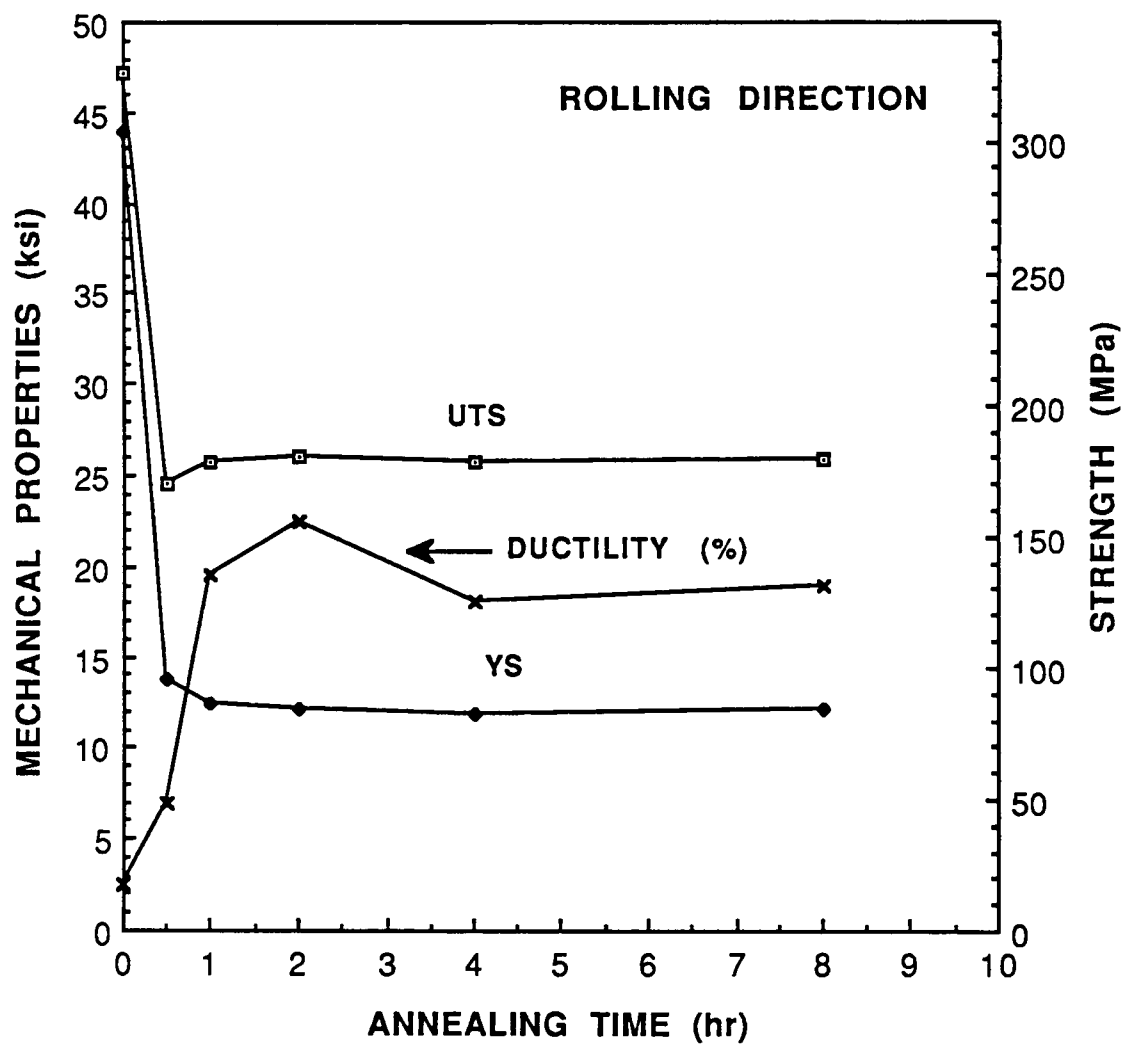


Figure 43. Mechanical properties in the rolling direction for 88% cold work material annealed at 288 °C (550 °F).

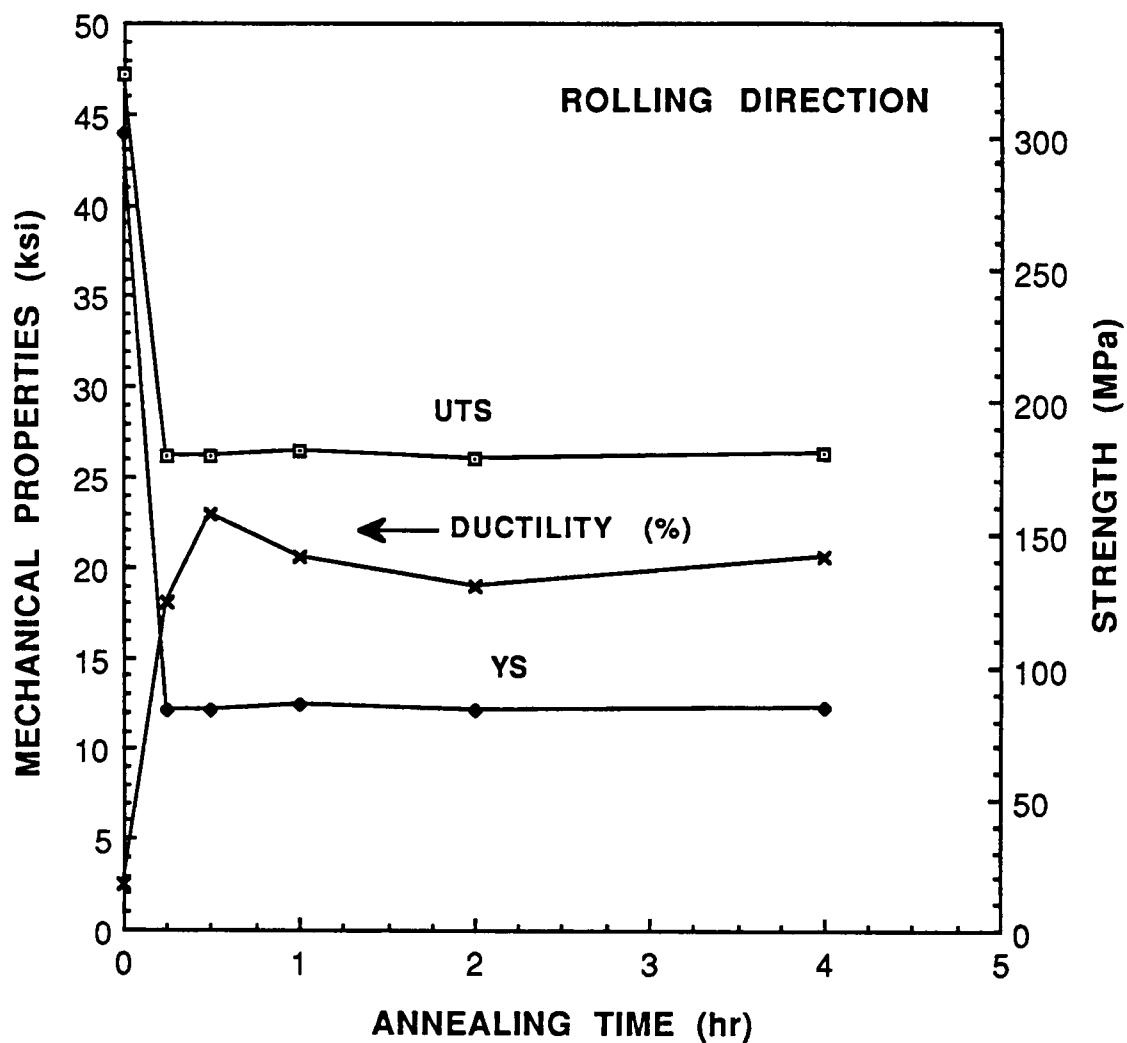


Figure 44. Mechanical properties in the rolling direction for 88% cold work material annealed at 316 °C (600 °F).

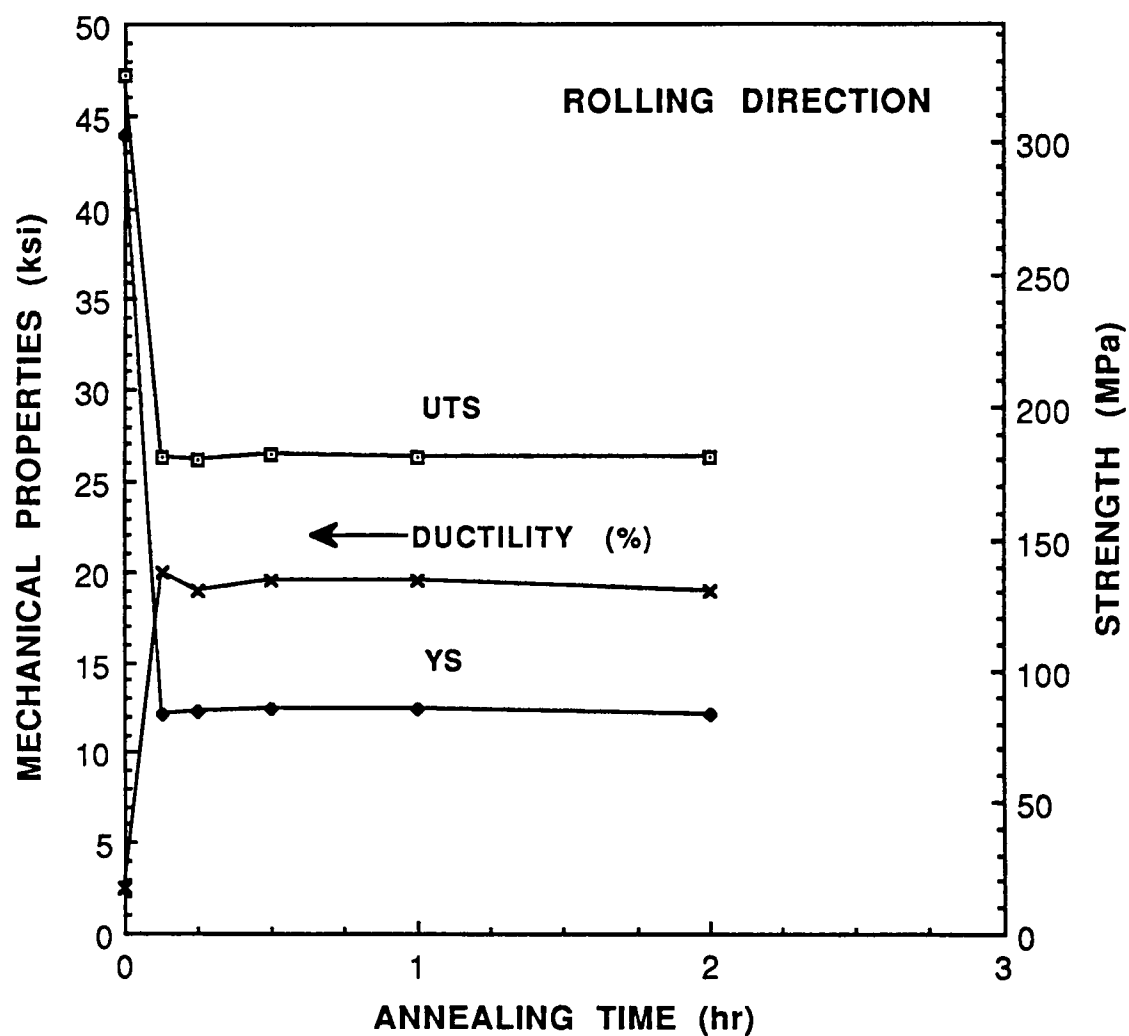
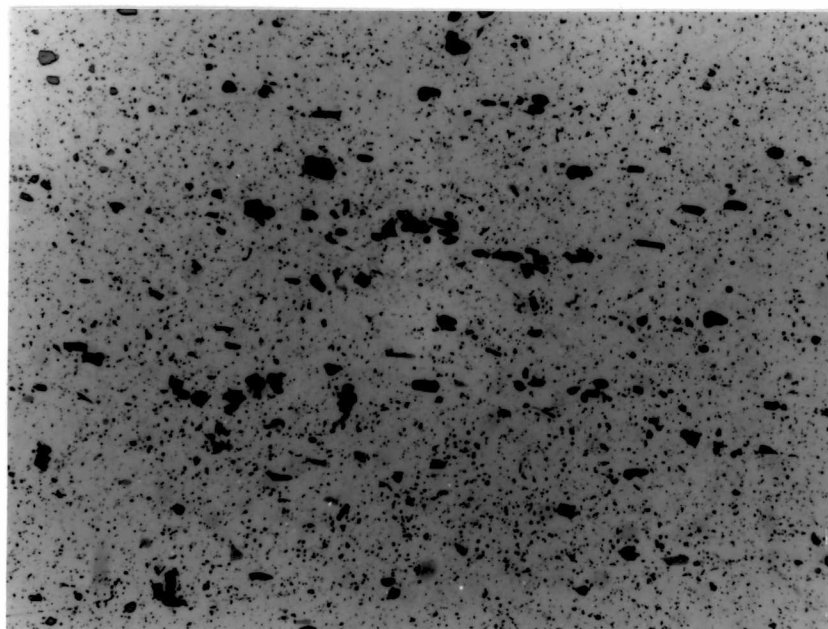
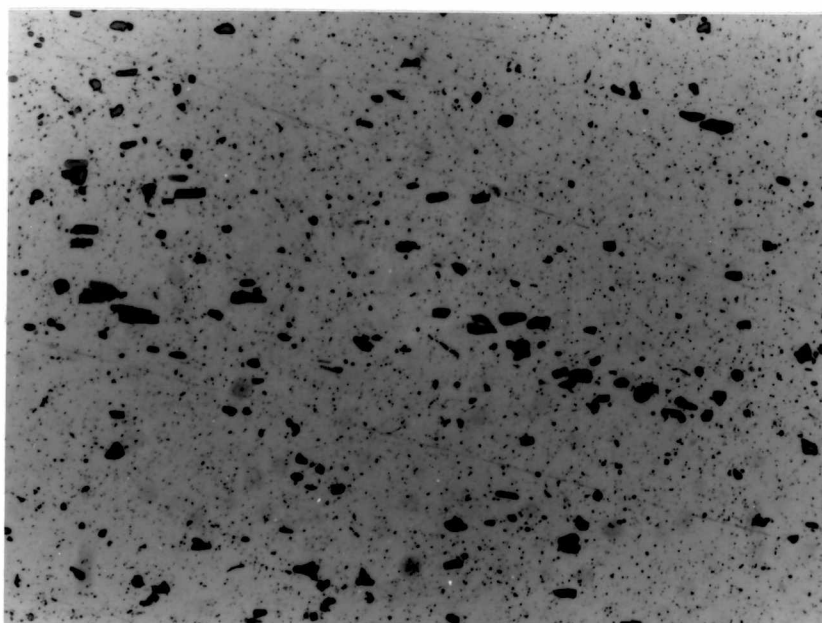


Figure 45. Mechanical properties in the rolling direction for 88% cold work material annealed at 332 °C (630 °F).



a) HOT ROLLED ROLLING DIRECTION →



b) SLOW ANNEAL ROLLING DIRECTION →

OM

50 μm

Figure 46. Microstructure at the rolling surface of hot rolled and slow anneal condition. As polished. 200x.

completely formed from the AlMn₆ particles. This was determined through analysis of many particles. The smaller particles were examined and determined to be the α -dispersoid. After etching with HF, there was no appreciable difference in microstructure.

Grain structures were evaluated only in the plane containing the longitudinal direction (see Figure 21). The grain structure of the hot rolled condition is shown in Figure 47. At the surface edge, the material is partially recrystallized. Toward the center, the material is not recrystallized and resembles a cold worked structure. This is the result of too low rolling temperatures and/or reduction per pass. If an annealing step does not follow hot rolling, variations in the recrystallized structure could cause variations in earing of the final product [15]. Thus, the hot rolled samples were heated at 13 °C/hour (25 °F/hour) to 332 °C (630 °F) and held for 2 hours, then water quenched. Figure 48 represents the grain structure of the annealed condition, showing large recrystallized grains.

After annealing, the samples were cold rolled. Figure 49 shows the cold work structures at 20%, 60%, and 88%. At 20% cold work (Figure 49 a), the grains are becoming slightly elongated. As the amount of deformation increases, the grains become progressively more elongated and aligned along the rolling direction (Figure 49 b and c).

The micrographs of the grain structures at 20% cold work after annealing are shown in Figures 50-52. The effect of annealing at 288 °C (550 °F) is shown in Figure 50 b-f. There is only a slight change in microstructure at increasing annealing times. In Figure 50 f, the microstructure changes with the growth of a few selective grains, showing



a) CENTER

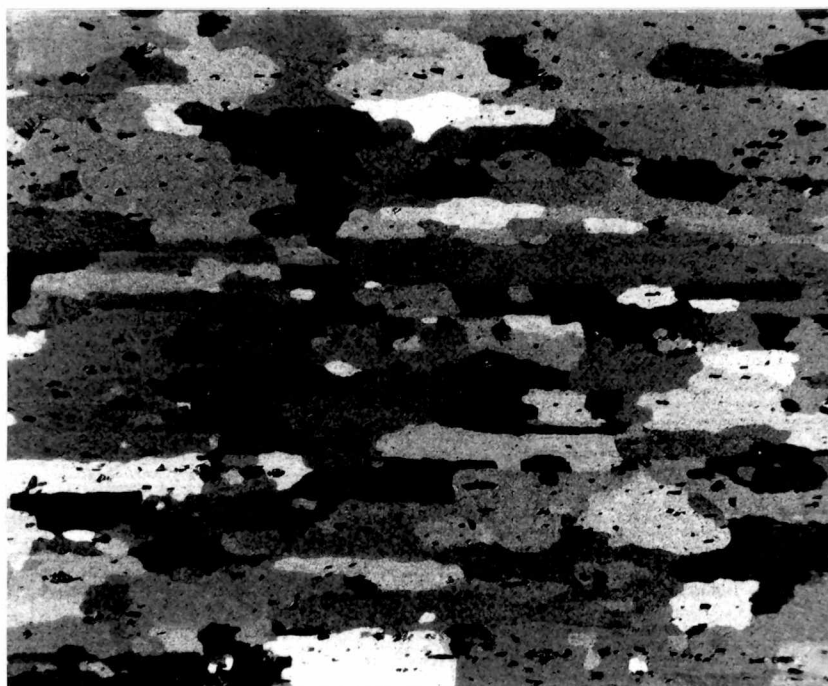


b) SURFACE EDGE

OM

100 μm

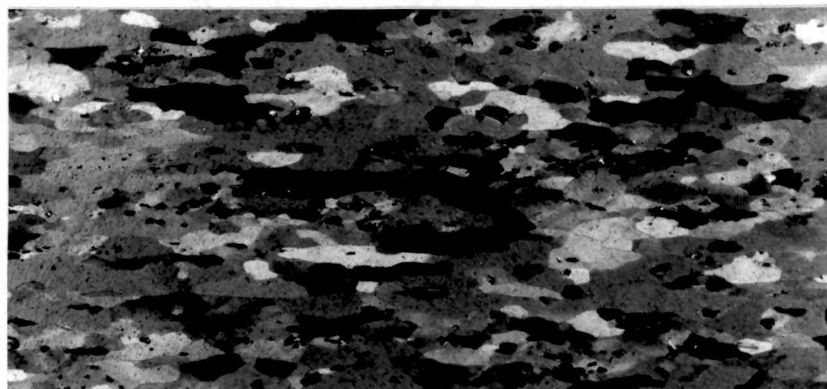
Figure 47. Grain structure in the rolling direction of hot rolled condition. Anodized with Barker's reagent. 200x.



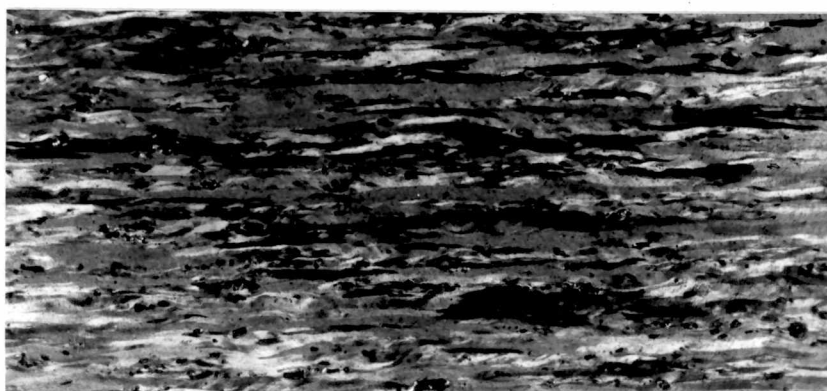
OM

100 μm

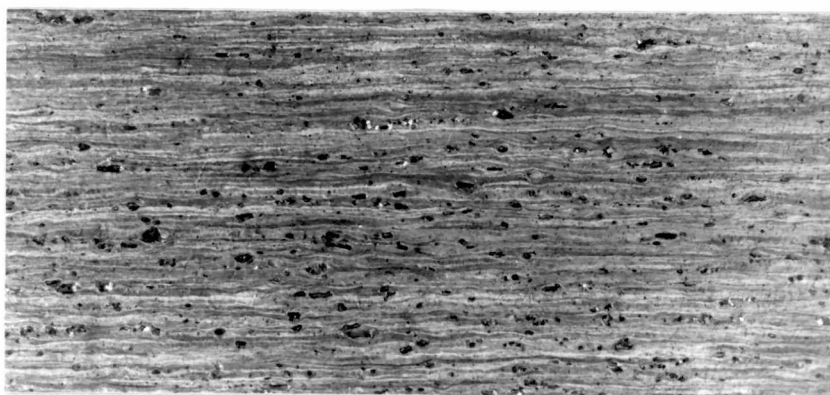
Figure 48. Grain structure in the rolling direction of slow anneal condition. Anodized with Barker's reagent. 200x.



a) 20% COLD WORK



b) 60 % COLD WORK

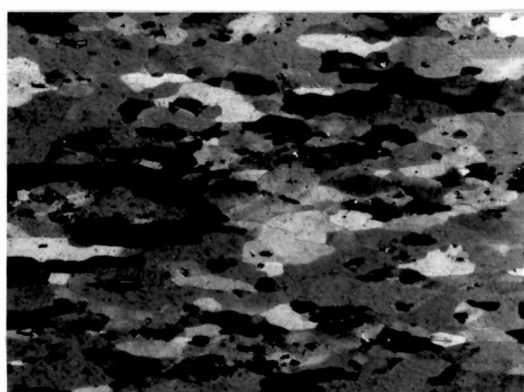


c) 88% COLD WORK

OM

100 μm

Figure 49. Grain structure in the rolling direction of 20%, 60%, and 88% cold work material. Anodized with Barker's reagent. 200x.



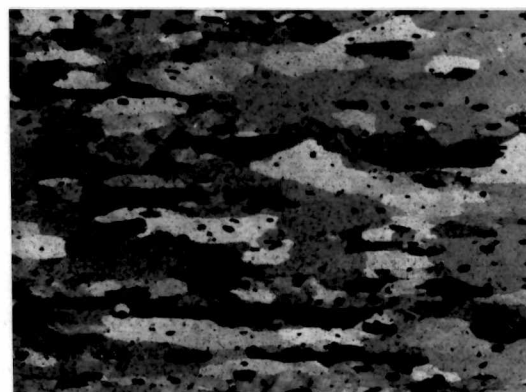
a) 20% Cold work



b) Annealed - 1/2 hr



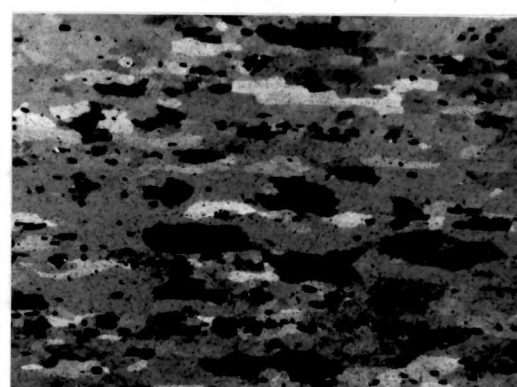
c) Annealed - 1 hr



d) Annealed - 2 hr



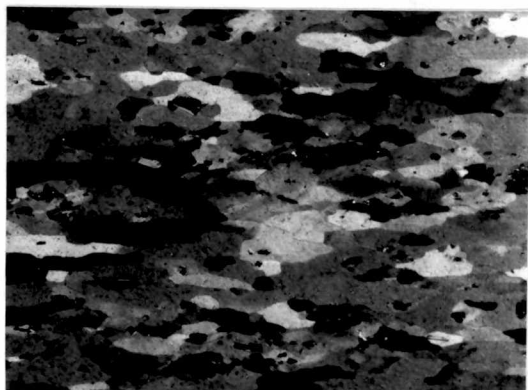
e) Annealed - 4 hr



f) Annealed - 8 hr

OM**100 μm**

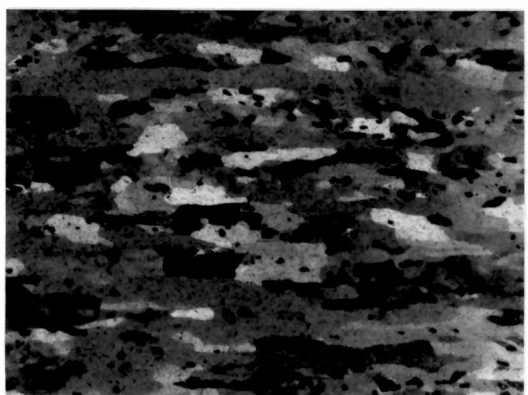
Figure 50. Grain structure of 20% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.



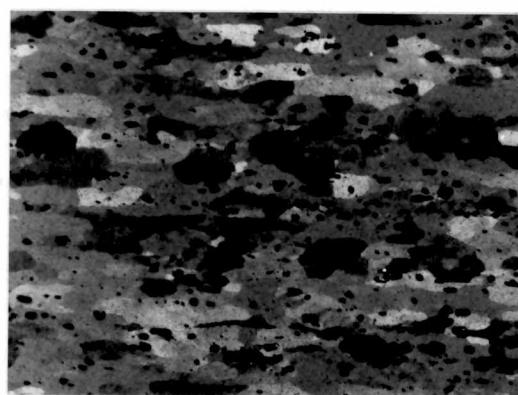
a) 20% Cold work



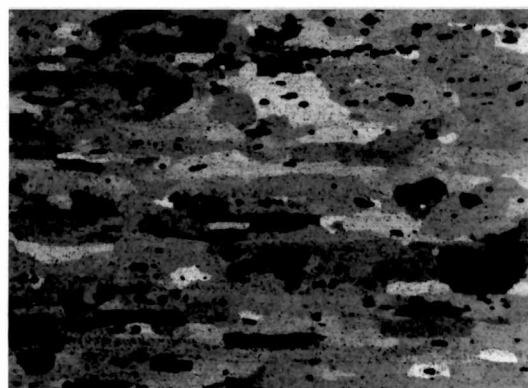
b) Annealed - 1/4 hr



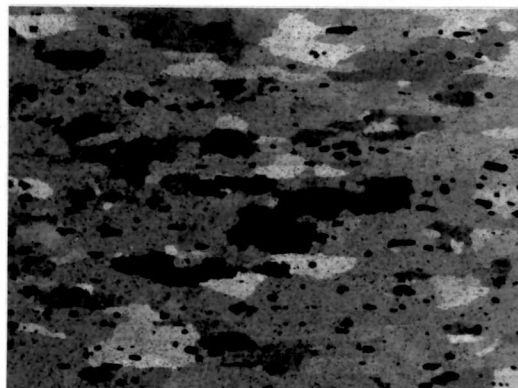
c) Annealed - 1/2 hr



d) Annealed - 1 hr



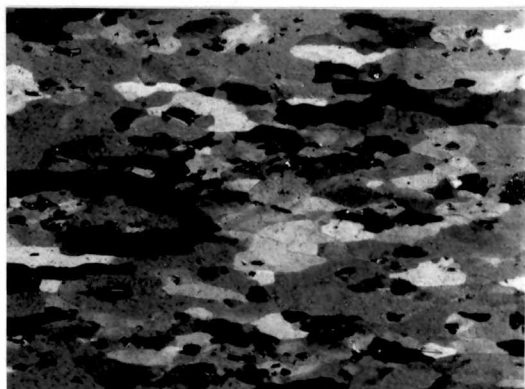
e) Annealed - 2 hr



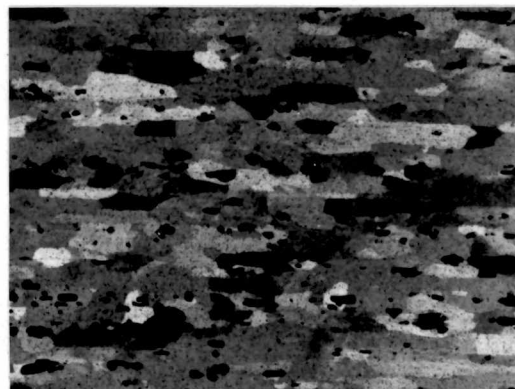
f) Annealed - 4 hr

OM**100 μ m**

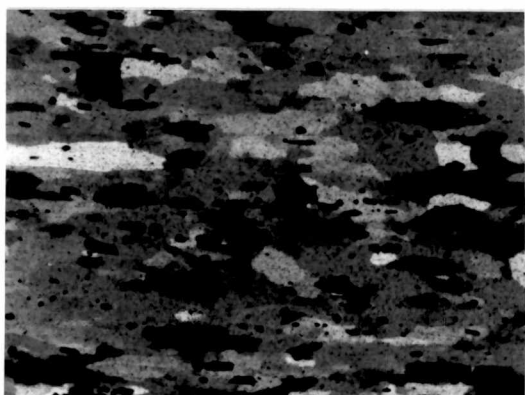
Figure 51. Grain structure of 20% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.



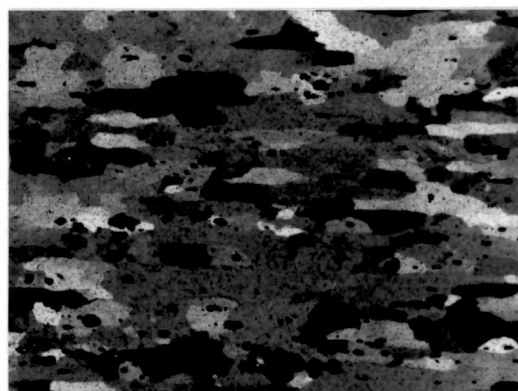
a) 20% Cold work



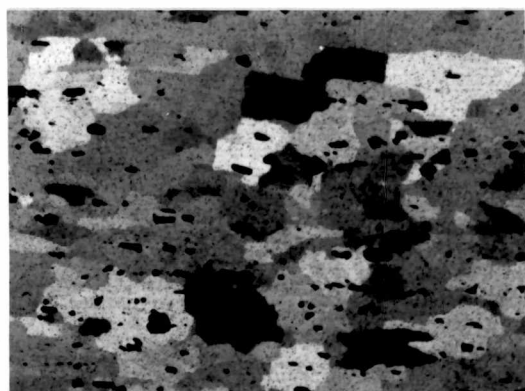
b) Annealed - 1/8 hr



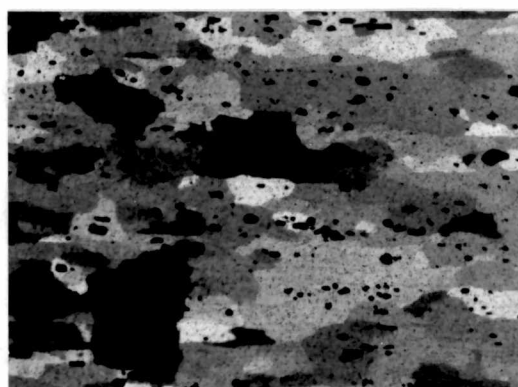
c) Annealed - 1/4 hr



d) Annealed - 1/2 hr



e) Annealed - 1 hr



f) Annealed - 2 hr

OM

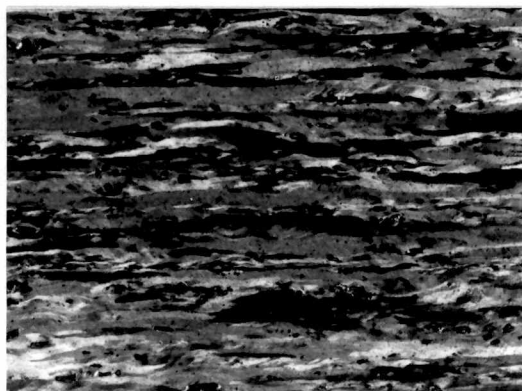
100 μm

Figure 52. Grain structure of 20% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.

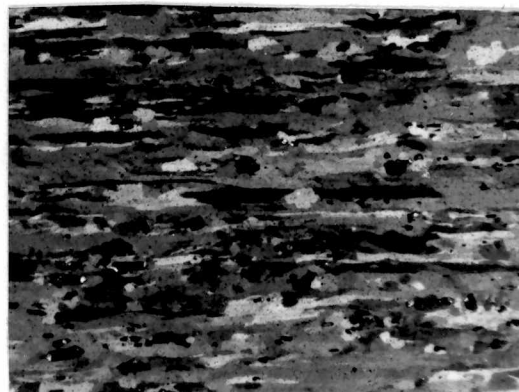
that start of recrystallization has occurred. The resultant elongated grains are due to the second phase particles. Figure 51 b-f represent the grain structure after annealing at 316 °C (600 °F). Selective growth of a few grains occurs but complete recrystallization does not. Upon annealing at 332 °C (550 °F), complete recrystallization occurs after 2 hours. This is shown in Figure 52 f. At the lower annealing times (Figure 52 b-d), the grains are slightly elongated. The grains, after complete recrystallization, are not as elongated.

The effect on grain structures after annealing 60% cold worked material is shown in Figures 53-55. In Figure 53, the material has been annealed at 288 °C (550 °F) for up to 8 hours. Partial recrystallization has occurred at 1/2, 1, and 2 hours of annealing, as shown in Figure 53 b-d, respectively. After 4 hours, complete recrystallization has occurred. The grain size is smaller and equiaxial compared to Figure 52 f of annealed, 20% cold work. Complete recrystallization after annealing at 316 °C (600 °F) is evident in Figures 54 b-f. The final recrystallized grain size remains relatively constant with increasing annealing temperature. Figure 55 b-f shows the recrystallized grain structure after annealing at 332 °C (630 °F). There is no apparent change in grain size after annealing 60% cold work specimens.

The grain structures of 88% cold work specimens after annealing are shown in Figures 56-58. Figure 56 shows the grain structure after annealing at 288 °C (550 °F). Partial recrystallization occurs after 1/2 hour (Figure 56 b). Complete recrystallization is shown in Figure 56 c-f after annealing 1 to 8 hours. The time required for complete recrystallization decreases with increasing reduction due to increased nucleation sites. Complete



a) 60% Cold work



b) Annealed - 1/2 hr



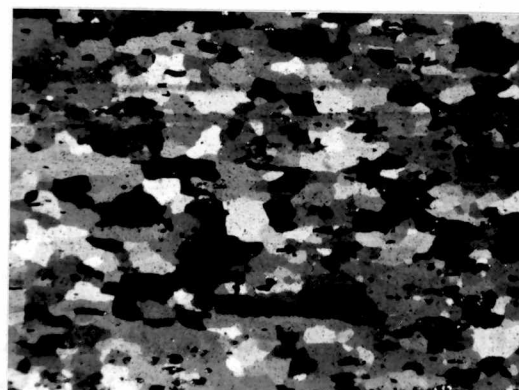
c) Annealed - 1 hr



d) Annealed - 2 hr



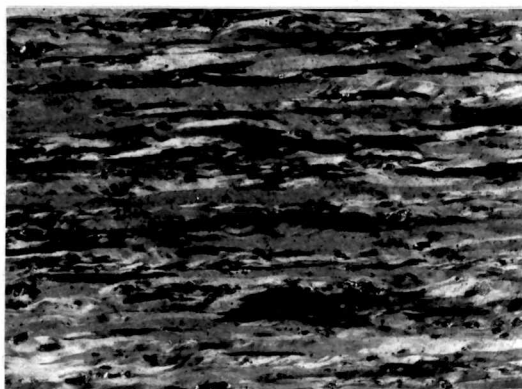
e) Annealed - 4 hr



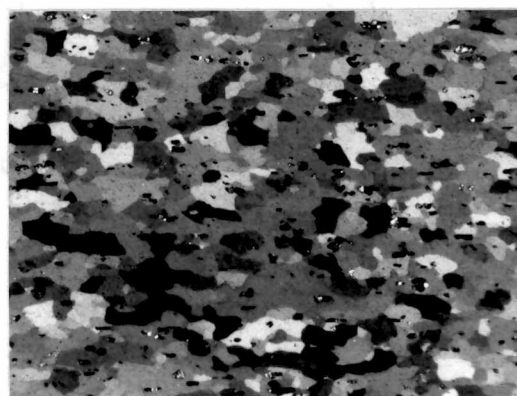
f) Annealed - 8 hr

OM100 μm

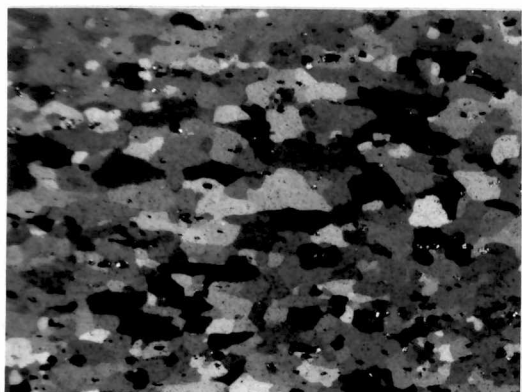
Figure 53. Grain structure of 60% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.



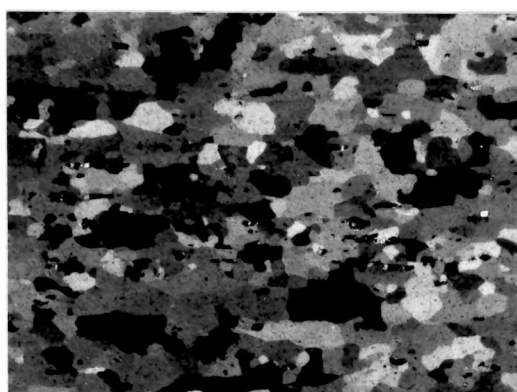
a) 60% Cold work



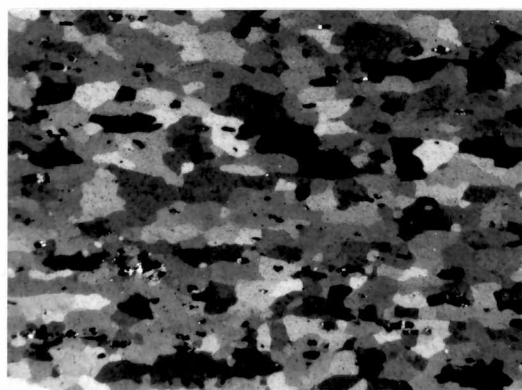
b) Annealed - 1/4 hr



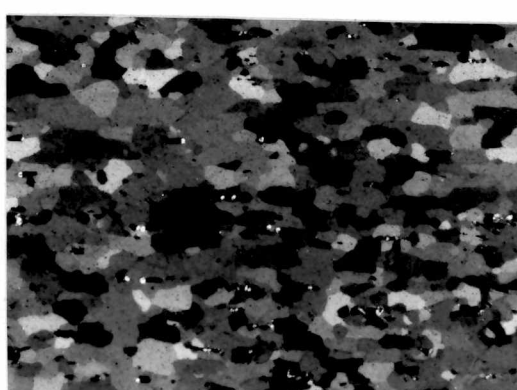
c) Annealed - 1/2 hr



d) Annealed - 1 hr



e) Annealed - 2 hr

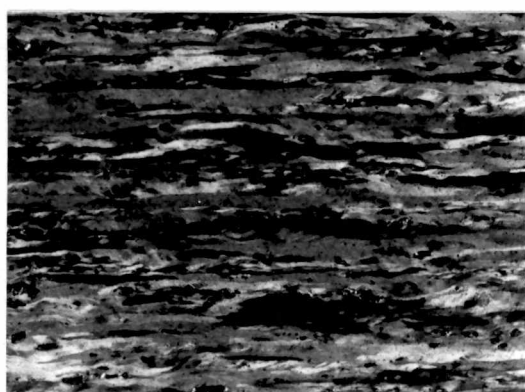


f) Annealed - 4 hr

OM

100 μm

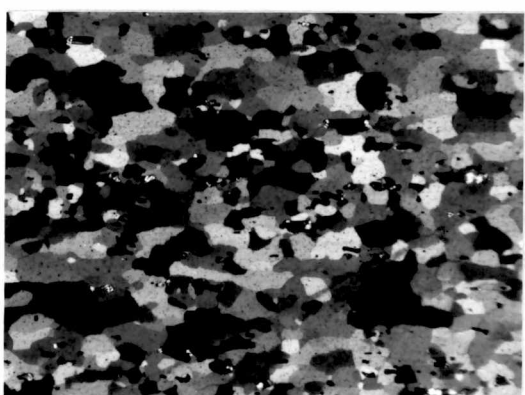
Figure 54. Grain structure of 60% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.



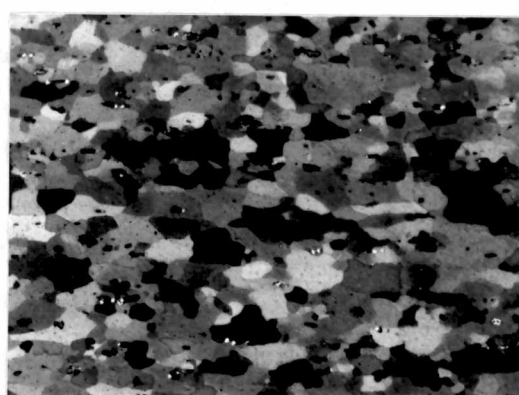
a) 60% Cold work



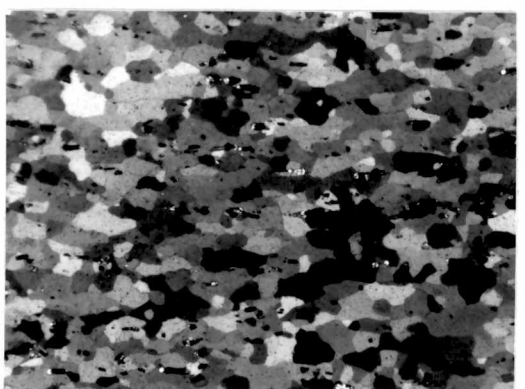
b) Annealed - 1/8 hr



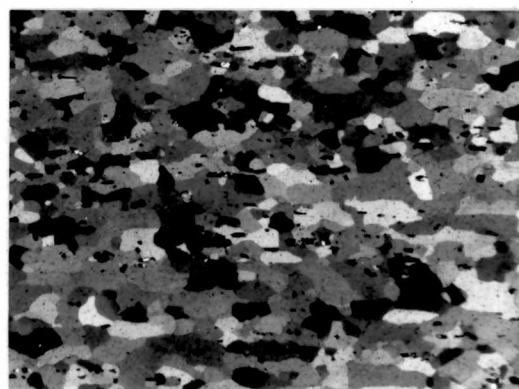
c) Annealed - 1/4 hr



d) Annealed - 1/2 hr



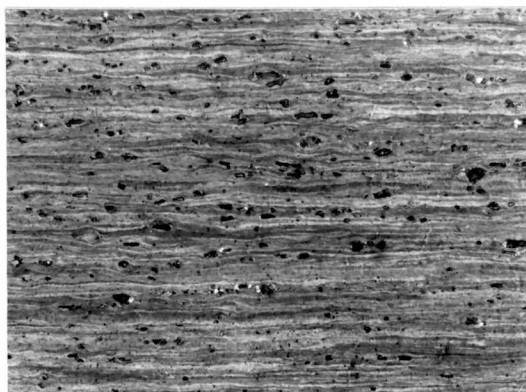
e) Annealed - 1 hr



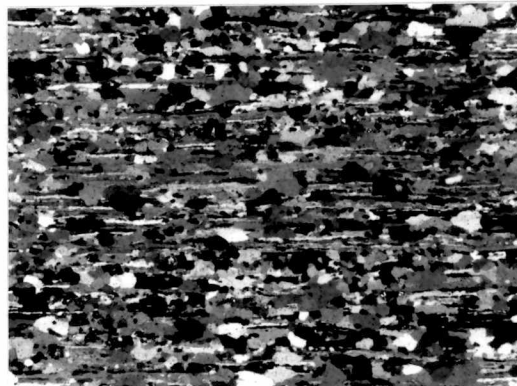
f) Annealed - 2 hr

OM**100 μ m**

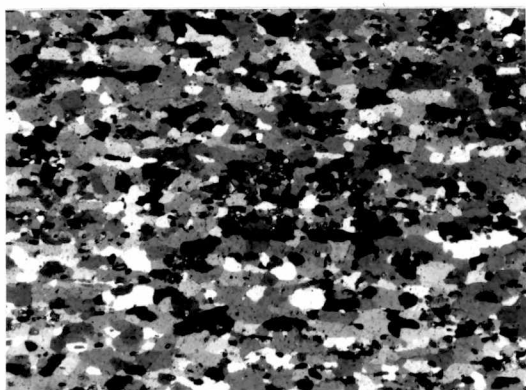
Figure 55. Grain structure of 60% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.



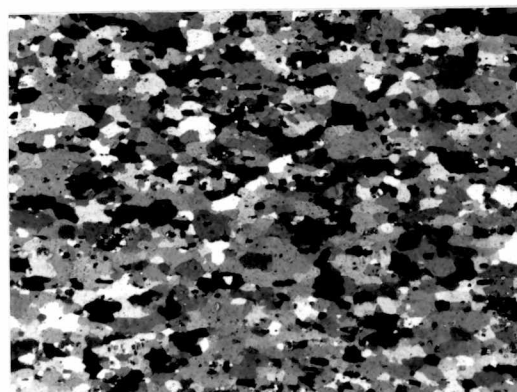
a) 88% Cold work



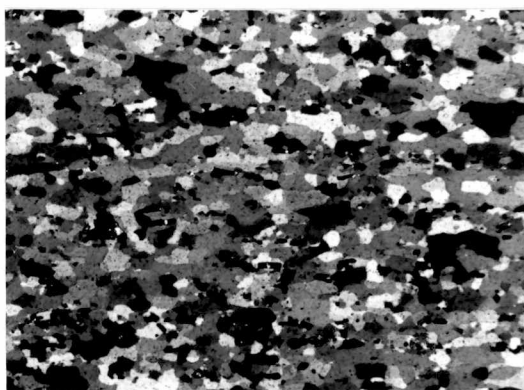
b) Annealed - 1/2 hr



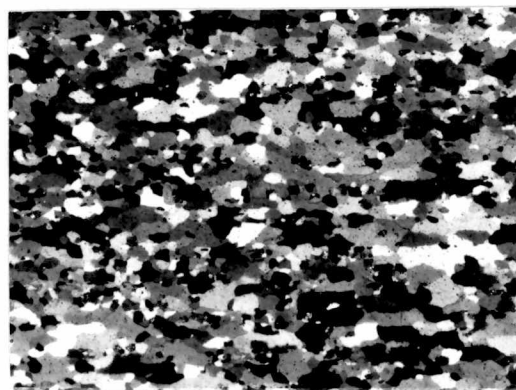
c) Annealed - 1 hr



d) Annealed - 2 hr



e) Annealed - 4 hr



f) Annealed - 8 hr

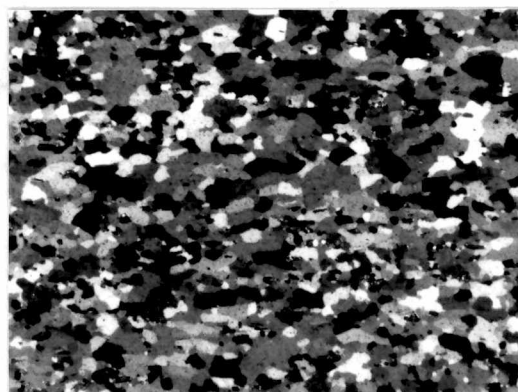
OM

100 μm

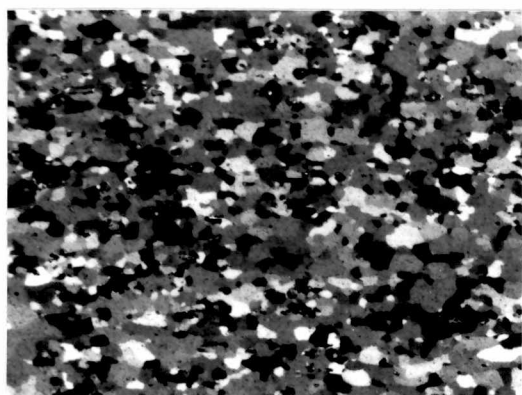
Figure 56. Grain structure of 88% cold work material annealed at 288 °C (550 °F). Anodized with Barker's reagent. 200x.



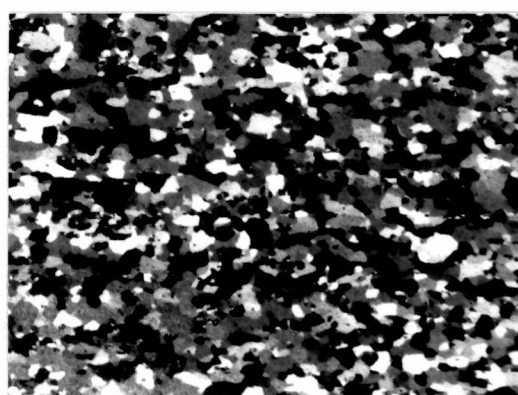
a) 88% Cold work



b) Annealed - 1/4 hr



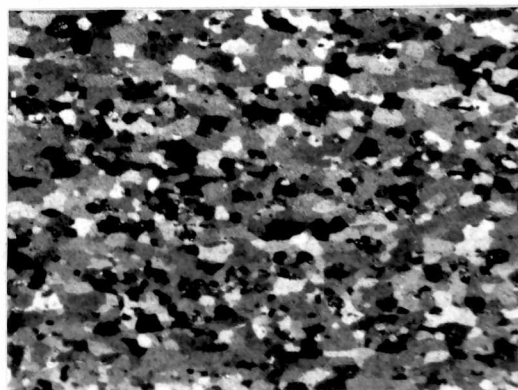
c) Annealed - 1/2 hr



d) Annealed - 1 hr



e) Annealed - 2 hr



f) Annealed - 4 hr

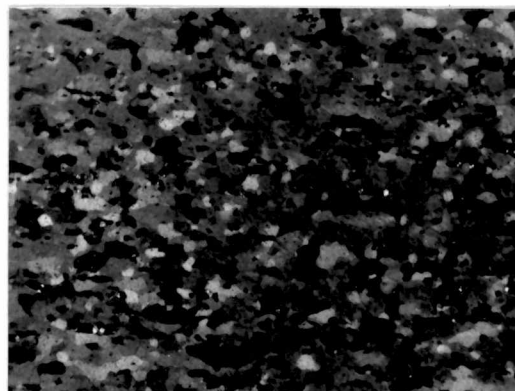
OM

100 μm

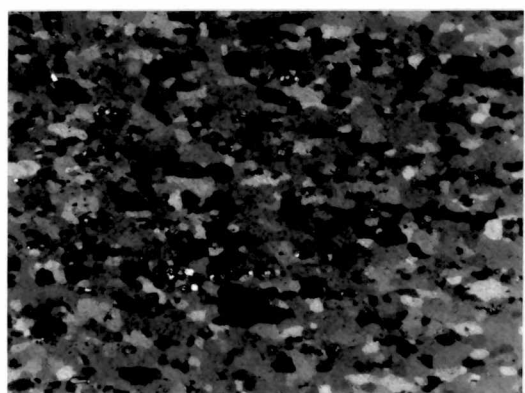
Figure 57. Grain structure of 88% cold work material annealed at 316 °C (600 °F). Anodized with Barker's reagent. 200x.



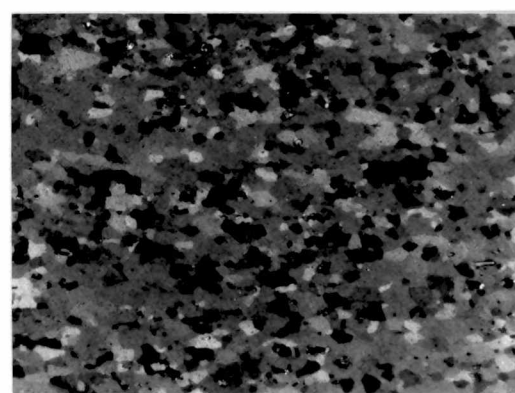
a) 88% Cold work



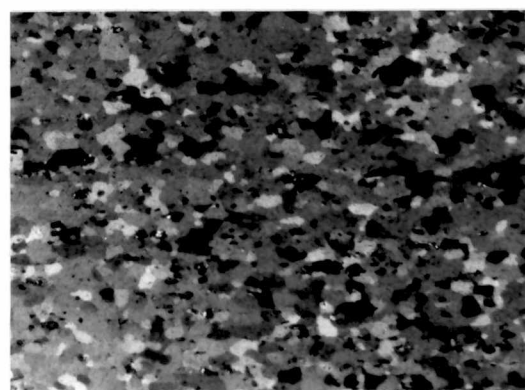
b) Annealed - 1/8 hr



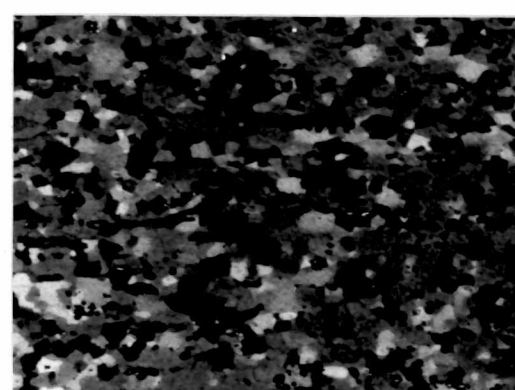
c) Annealed - 1/4 hr



d) Annealed - 1/2 hr



e) Annealed - 1 hr



f) Annealed - 2 hr

OM

100 μm

Figure 58. Grain structure of 88% cold work material annealed at 332 °C (630 °F). Anodized with Barker's reagent. 200x.

recrystallization occurs after 1/4 hour at 316 °C (600 °F) as shown in Figure 57 b-f. The grain structure after annealing at 332 °C (630 °F) is shown in Figure 58. Recrystallization is complete after 1/8 hour (Figure 58 b-f).

The nucleation sites available for recrystallization increase with increasing percent cold work, so it is expected that the recrystallized grain size will decrease with increasing reduction. Grain size measurements were made on the recrystallized material in Figures 52-58. Table 4 shows the grain size for each condition. Grain size decreases (grain size number increases) with increasing percent reduction. The annealing temperature for 20%, 60%, and 88% cold work had little effect on the final size of the recrystallized grains. Morris and Duggan [25] also observed this in their grain size after annealing at 425 °C- 450 °C (797 °F - 842 °F). Also, the grain size change had little effect on the mechanical properties.

TEXTURING

Texturing is the arrangement of grains in a certain crystallographic orientation. There are two primary texturing conditions in sheets: rolling (deformation) texture and recrystallization (annealing) texture. These textures cause ears, wavy edges along top of cups, to form during deep drawing. The position of the ears indicates the dominating texture. In the annealed condition, ears forming at 45° to rolling direction indicates that the retained R- texture is dominating, while ears at 0° and 90° to rolling show that the cube texture is dominating (Figure 13).

X-ray diffraction and deep drawing of cups were used to determine the crystallographic texturing for cold working and final anneal at 332 °C

Table 4. ASTM grain size of completely recrystallized material.

Temperature °C (°F)	Time (hr)	% Cold Work		
		20%	60%	88%
288 °C (550 °F)	1	-----	-----	11.6
	2	-----	-----	11.3
	4	-----	8.1	11.3
	8	-----	8.1	11.2
316 °C (600 °F)	1/4	-----	7.9	11.5
	1/2	-----	7.9	11.4
	1	-----	7.7	11.7
	2	-----	8.0	11.3
	4	-----	7.9	11.5
332 °C (630 °F)	1/8	-----	8.4	11.2
	1/4	-----	8.2	11.2
	1/2	-----	8.0	11.1
	1	-----	8.1	11.2
	2	6.2	8.1	11.5

(630 °F). A correlation between normalized relative integrated intensities and earing after deep drawing was evaluated.

The intensity of the {200} peaks was determined and taken as a relative measurement of the intensity of the cube oriented grains. Table 5 shows the integrated intensities for the samples which were hot rolled, annealed, and then cold worked. The cube intensity increases after slow annealing, which indicates a high cube texture has formed. At increasing amounts of deformation, the cube intensity decreases and the earing changes from 90° to 45° to the rolling direction. Table 6 shows the percent earing after deep drawing. Samples in the hot rolled and slow annealed conditions could not be drawn due to thickness limitations. Below 60% reduction, 90° earing (cube texture) occurs, and above 60%, 45° earing (rolling texture) occurs. At 60% cold work, the texture is balanced resulting in no earing (Figure 13 d). Thus, this is a transition point between the cube and rolling textures.

X-ray analyses were taken for both the {220} and {200} planes after annealing for 20%, 60%, and 88%. The high intensity of a {220} peak indicates the presence of deformation texture components. This was examined to determine any changes in intensity of these planes on annealing. Table 7 shows the normalized x-ray intensities from the tensile specimens. The cube intensity decreased after annealing the 20% cold work material, while it increased for the annealed samples at 60% and 88% reductions. The rolling oriented plane intensity, {220}, for 20% cold work material decreased after 1 to 2 hours of annealing. For the 60% and 88%

Table 5. X-ray diffraction results of {200} peak for cold work material.

Condition	Peak Integral	* Normalized
Random	100059	1.0
Hot Rolled	165739	1.7
Slow Anneal	378593	3.8
Cold Work - 12%	350387	3.5
- 20%	313878	3.1
- 40%	211688	2.1
- 60%	161429	1.6
- 80%	92831	0.9
- 88%	83421	0.8
- 92%	82855	0.8
- 96%	49769	0.5

* peak integral of sample/random sample

Table 6. Deep drawing results of earing (%) for cold work material.

Cold work (%)	Earing (%)	Direction
20	4.5	0° - 90°
60	0	no earing
80	7	45°
88	5.5	45°
92	3.5	45°

Table 7. X-ray diffraction results from tensile specimens of {200} and {220} peaks (normalized) after annealing at 332 °C (630 °F).

Condition	% Cold work					
	{200} peak			{220} peak		
	20	60	88	20	60	88
Cold work	3.1	1.6	0.8	2.1	2.1	2.6
Annealed - 1/8 hour	2.6	2.6	3.3	2.2	1.6	1.9
Annealed - 1/4 hour	2.5	2.3	3.3	2.2	1.3	2.0
Annealed - 1/2 hour	2.2	2.6	3.0	2.0	1.5	1.7
Annealed - 1 hour	2.6	2.5	3.1	1.6	1.7	1.9
Annealed - 2 hour	2.4	2.4	3.2	1.6	1.7	1.8

reductions this intensity decreased after annealing and remained relatively constant.

Table 8 shows the normalized x-ray intensities from the deep drawn cups for 60% and 88% annealing studies. These samples were annealed at a slower heat-up rate than for the tensile specimens. A change in x-ray intensities occurred. The intensity of the {220} planes increased from the cold work value after annealing. The results from the tensile specimens showed a decrease. For the 60% cold work samples, a change in texture is observed between the tensile specimens (fast heat-up rate) and deep drawn cups (slow heat-up rate). The x-ray data from the tensile specimens predicted that earing would occur at 90° to the rolling direction, but the results from the x-ray data of the cups indicate 45° earing. The earing tests showed earing at 45° to the rolling direction.

The percent earing results from drawing tests are shown in Table 9. The percent earing of the 20% cold work material is 4.5% and occurs at 0° and 90° to the rolling direction. This cube texture also predominates after annealing and percent earing remains relatively constant at 5%. The retained cube texture would be expected due to the initial cube texture prior to annealing.

The 60% cold work samples showed an unexpected change in earing after annealing. Initially, the material in the cold worked condition had no earing. After annealing 1/8 hour, the earing became mixed (Figure 13 c) and then with further annealing changed to a high 45° earing (Table 9). The results of the x-ray analyses would indicate the material should have a cube texture. Heat-up rates do have an effect on the texture generated after

Table 8. X-ray diffraction results from deep drawn cups of {200} and {220} peaks (normalized) after annealing at 332 °C (630 °F).

Condition	% Cold work			
	{200} peak		{220} peak	
	60	88	60	88
Cold work (Tensile)	1.6	0.8	2.1	2.6
Annealed - 1/8 hour	2.0	5.3	3.8	4.6
Annealed - 1/4 hour	4.4	3.6	5.3	3.5
Annealed - 1/2 hour	2.7	3.9	3.3	4.5
Annealed - 1 hour	4.3	3.3	4.9	3.6
Annealed - 2 hour	3.7	4.2	3.6	4.3

Table 9. Deep drawing results of earing (%) for annealed material.

Cold work (%)	Time (hr) at 332 °C (630 °F)	Earing (%)	Direction
20	0	4.5	90°
	1/8	6.0	90°
	1/4	4.5	90°
	1/2	5.0	90°
	1	6.0	90°
	2	4.5	90°
60	0	0	no earing
	1/8	1.5	mixed
	1/4	6.0	45°
	1/2	6.0	45°
	1	6.0	45°
	2	6.0	45°
88	0	5.5	45°
	1/8	2.0	90°
	1/4	2.5	90°
	1/2	bad test	----
	1	2.5	90°
	2	2.0	90°

recrystallization. The tensile specimens were annealed in a salt bath (higher heat-up rate) while the blanks for drawing were heated in an air furnace (slower heat-up rate). This difference in heat up rate could have an effect on precipitation of dispersoids resulting in a change in texturing properties. For a complete understanding of this difference, these conditions should be evaluated through transmission electron microscopy (TEM) to determine their effect on texturing behavior.

The cold work material at 88% reduction had earing of 5.5 % at 45° to rolling direction. (A stabilize anneal at 149 °C (300 °F) was given prior to drawing to increase its drawability. This could reflect a decrease in the percent earing compared to the earing in the 80 % cold work cup. (see Table 6)). Annealing caused small earing of approximately 2 % at 0° and 90° to the rolling direction. The high rolling texture prior to annealing reduced the final percent earing after recrystallization compared with the annealed 20% cold work material.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The results from this research project yield the following conclusions:

- 1) A decrease in ultimate tensile and yield strength with a increase in ductility upon annealing revealed the hot rolled material was not completely recrystallized.
- 2) Specimen orientation showed very little variation in ultimate tensile strength, yield strength, and ductility resulting in low anisotropic behavior in these properties.
- 3) Prior deformation changes the time and temperature for recrystallization. Increasing reductions decreases the time and temperature necessary for complete recrystallization.
- 4) The recrystallization temperature for 20% cold work material was 332 °C (630 °F) in 2 hours. For 60% and 88% cold work material the temperature for complete recrystallization was 316 °C (600 °F) in 1/4 hour.
- 5) The amount of deformation does not affect the mechanical properties after annealing. Mechanical properties remain relatively constant after recrystallization.
- 6) Although, grain size decrease with increasing reduction, the mechanical properties remained relatively constant.

- 7) Annealing temperature does not effect the final mechanical properties after recrystallization.
- 8) Increasing reductions decrease the final recrystallized grain size. At constant reductions, the grain size does not change with annealing temperature. Therefore, the final grain size could be a function of interparticle spacing in particle stimulated nucleation.
- 9) Complete recrystallization, revealed through microstructural examination, is consistent with the results from mechanical properties.
- 10) X-ray diffraction analysis for the cold worked samples showed good correlation with the deep drawing tests.
- 11) X-ray diffraction analysis of the annealed samples and the drawing tests did not show good correlation in predicting the texture. However, the magnitude of the retained rolling texture was not evaluated.
- 12) Heat-up rate does have an effect on the texturing behavior after annealing. This is shown through x-ray diffraction.
- 13) Heat-up rates have an effect on the material at the transition point (between cube and rolling textures). The transition point for this material occurred at 60% reduction.

RECOMMENDATIONS

Recommendations for future work include a better correlation between x-ray analyses and deep drawing tests after annealing. The prediction of the predominating texture from x-ray analyses is important. A possible solution

would be to determine pole figures at various stages. This would give the spread for the predominating orientation(s) and allow predictions for the relative x-ray intensities. The relative x-ray intensities would enable a quick and reliable way of quality control of the material throughout manufacturing process.

Particle distribution plays an important role in the recrystallization kinetics as discussed in the literature. The grain structure depends largely on particle stimulated recrystallization. The effect that large primary particles has on recrystallization should be examined. The effect of heat-up rate during annealing on the change in dispersoid density needs to be examined. Also, the recrystallization kinetics due to dispersoid precipitation needs to be determined. Both the effect of large primary particles and dispersoids should be evaluated to determine their role in the final texture.

Microstructural evaluation of samples at additional annealing temperatures and times should be conducted. This would allow the recrystallization kinetics, primarily the activation energy, to be calculated through time-temperature plots.

LIST OF REFERENCES

LIST OF REFERENCES

1. I. J. Polmear, *Light Alloys Metallurgy of the Light Metals*, American Society for Metals, Metals Park, Ohio (1981). pp. 59.
2. A. J. McAllister and J. L. Murray, The (Al-Mn) Aluminum-Manganese System, *Bulletin of Alloy Phase Diagrams* 8:438 (1987).
3. I. J. Polmear, *ibid.* pp. 70.
4. American Society for Metals, Metallography Structures and Phase Diagrams, *Metals Handbook*, 8th ed., Metals Park, Ohio (1974). pp. 261.
5. The Aluminum Association's Task Group on Segregation in Thick Sections, Macrosegregation in Wrought Aluminum Alloys, *Light Metal Age* 44:5-9 (1986).
6. H. Hero and S. E. Naess, Stretch formability of Al-Mn alloys, *Metals Technology* 6:245-251 (1979).
7. P. Furrer and G. Hausch, Recrystallization behaviour of commercial Al-1% Mn alloy, *Metal Science* 13:155-162 (1979).
8. E. J. Westerman and D. I. Smith, Effects of preheating on 3104 alloys ingot microstructure, *Kaiser Aluminum Research Report CFT RR 86-6*, May 30, 1986.
9. P. Furrer, Controlling the annealing behaviour and grain size of aluminium alloys, (reference unknown).
10. H. D. Merchant, T. Z. Kattamis, and G. Scharf, Homogenization of aluminum alloys, *Homogenization and Annealing of Aluminum and Copper Alloys*, The Metallurgical Society (1988). pp. 1-52.
11. D. J. Lloyd, The influence of particles and deformation structure on recrystallization, *Microstructural Control in Aluminum Alloys: Deformation, Recovery, and Recrystallization*, The Metallurgical Society, Inc., New York (1985). pp. 45-66.
12. L. J. Cartmell, F. King, and G. Salt, Hot rolling of sheet and strip: aluminium and aluminium alloys, *Metals Technology* 2:313-317 (1975).

13. R. T. Thorley and G. E. G. Tucker, The control of earing in aluminium and its alloys, *Journal of the Institute of Metals* 86:353-361 (1957-58).
14. A. Karlsson , A. Oscarsson, B. Lehtinen, and W. Hutchinson, Influence of homogenisation on structure and earing in aluminium alloy 3004, *Homogenization and Annealing of Aluminum and Copper Alloys*, The Metallurgical Society (1988). pp. 99-117.
15. J. C. Blade, Recrystallization of hot rolling of dilute aluminium alloys, *Metal Science* 13:206-210 (1979).
16. K. J. Gardner and R. Grimes, Recrystallization during hot deformation of aluminium alloys, *Metal Science* 13:216-222 (1979).
17. J. C. Blade A. J. Bryant, and A. T. Thomas, Alloy composition and microstructure control in relation to mechanical working for aluminium alloys, *Metals Technology* 3:380-386 (1976).
18. F. R. Castro-Fernandez and C. M. Sellars, Static recrystallization and recrystallisation during hot deformation of Al-1Mg-1Mn alloy, *Material Science and Technology* 4:621-627 (1988).
19. J. R. Cotner and W. J. McG. Tergart, High-temperature deformation of aluminium-magnesium alloys at high strain rates, *Journal Institute of Metals* 97:73-79 (1969).
20. P. A. Hollinshead and T. Sheppard, Substructure morphology in aluminium alloys AA 3303 and AA 3004, *Materials Science and Technology* 3:1019-1024 (1987).
21. A. G. Guy and J. J. Hren, *Elements of Physical Metallurgy*, Addison-Wesley Publishing Company, Reading Massachusetts (1974). pp. 440.
22. D. J. Lloyd, Recrystallization of unhomogenized aluminium-manganese alloys, *Metal Science* 16:304-308 (1982).
23. F. J. Humphreys, Recrystallization mechanisms in two-phase alloys, *Metal Science* 13:136-145 (1979).
24. U. Koster, Recrystallization involving a second phase, *Metal Science* 8:151-160 (1974).
25. P. L. Morris and B. J. Duggan, Precipitation and recrystallization in Al-1.8%Mn alloy, *Metal Science* 12:1-7 (1978).

26. P. R. Mould and P. Cotterill, The effect of particle content and matrix grain size on the recrystallisation of two-phase aluminium-iron alloys, *Journal of Materials Science* 2:241-255 (1967).
27. F. J. Humphreys, The nucleation of recrystallization at second phase particles in deformed aluminium, *Acta Metallurgica* 25:1323-1344 (1977).
28. E. Nes, The effect of a fine particle dispersion on heterogeneous recrystallization, *Acta Metallurgica* 24:391-398 (1976).
29. N. Hansen and B. Bay, Initial Stages of recrystallization in aluminium containing both large and small particles, *Acta Metallurgica* 29:65-77 (1981).
30. G. Y. Chin, Textured Structures, Metallography Structures and Phase Diagrams, *Metals Handbook* 8th ed., American Society for Metals, Metals Park, Ohio (1974). pp.229-232.
31. Marc Van Lancker, *Metallurgy of Aluminium Alloys*, John Wiley and Sons, Inc., New York, New York (1967). pp. 175.
32. John. E. Hatch, *Aluminum properties and physical metallurgy*, American Society for Metals, Metals Park, Ohio (1984). pp. 127.
33. B. Anderson and E. Nes, Nucleation of recrystallization in a commercial AlMn-Alloy, 1st Ris Int Symposium on Metallurgy and Materials Science, Roskilde, Denmark (1980). pp. 115-119.
34. K. Kwag and J. G. Morris, The effect of structure on the mechanical behavior and stretch formability of constitutionally dynamic 3000 series aluminum alloys, *Materials Science and Engineering* 77:59-74 (1986).
35. R. Orsund and E. Nes, Effect of particles on recrystallization textures in aluminum-manganese alloys, *Scripta Metallurgica* 22:665-669 (1988).
36. R. Orsund and E. Nes, A model for the nucleation of recrystallization from particles: the texture aspect, *Scripta Metallurgica* 22:671-676 (1988).
37. R. Grimes, Grain control in aluminium, *Metal Science* 8:176-184 (1974).
38. E. Nes, Grain Size and texture control in commercial aluminum alloys, *Microstructural Control in Aluminum Alloys: Deformation, Recovery, and Recrystallization*, The Metallurgical Society, Inc., New York (1985). pp. 95-108.

39. K. Fukada, M. Mizouchi, and T. Kajiyama, Effect of heating rate and temperature on annealing textures of 3004 aluminum alloy, *SKY Aluminum Company Ltd.*, Japan (not dated).
40. E. Nes, Recrystallization in alloys with bimodal particle size distributions, 1st Ris Int Symposium on Metallurgy and Materials Science, Roskilde, Denmark (1980). pp. 85-95
41. R. A. Rida and W. B. Hutchinson, The origin of cube texture in copper, *Acta Metallurgica* 30:1929-1939 (1982).
42. A.L. Dons and E. Nes, Nucleation of cube texture in aluminium, *Materials Science and Technology* 2:8-18 (1986).
43. L. J. Barker, Revealing the grains structure of common aluminum alloy metallographic specimens, *Transactions ASM* 42:347-356 (1950).
44. American Society for Testing Materials, Standard methods for determining average grain size, Spec E 112, Philadelphia, Pennsylvania (1982).
45. L. Yi-Lin, L. Delaey, E. Aernoudt and O. Arkens, Substructure development and mechanical properties in cold-rolled aluminium alloy 3004 I: Substructure development, *Materials Science and Engineering* 96:125-137 (1987).
46. L. Yi-Lin, L. Delaey, E. Aernoudt and O. Arkens, Substructure development and mechanical properties in cold-rolled aluminium alloy 3004 II: Mechanical properties, *Materials Science and Engineering* 96:139-146 (1987).
47. I. R. Dover and J. D. Embury, The influence of microstructural features on the formability of aluminium alloys, *Materials Forum* 9:188-210 (1986).

APPENDIX

FURNACE CONTROL

The proportional band (PB) was lowered until oscillations in the temperature were observed with no rate or reset. At 1% proportional band no oscillations were observed; oscillations occurred only by adding rate and reset. By changing the PB to 45% and adding rate only, the temperature increased very slowly and never reached the set point temperature. Resetting the rate value to zero and adding a small amount of reset (<1.0 resets/minute) was effective, but a large overshoot in set point occurred. Decreasing the PB setting decreased the amount of overshoot of the set point temperature. Thus the final settings for controlling the furnace temperature were PB=39%, reset=0.40 resets/minute, and rate=0.

DATA FROM MECHANICAL PROPERTIES VS. PERCENT COLD WORK- (ksi)										
CONDITION	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
HOT ROLLED	29.1	27.7	29.1	21.9	21.4	22.9	21.0	21.0	18.0	
HOT ROLLED	29.2	27.6	28.8	22.0	20.7	22.5	22.0	20.0	18.0	
HOT ROLLED	29.3	27.6	29.0	21.7	21.0	22.0	21.0	20.0	18.0	
ANNEALED	24.7	24.7	24.4	12.1	12.7	12.8	23.0	26.0	26.0	
ANNEALED	24.8	24.6	24.3	12.7	13.1	12.4	25.0	28.0	27.0	
ANNEALED	24.8	24.7	24.3	12.9	12.8	12.9	27.0	30.0	27.0	
CW - 12	28.1	28.1	28.6	26.6	26.3	26.1	10.0	11.0	11.0	
CW - 12	28.1	27.9	28.7	26.4	25.9	26.2	10.0	11.0	11.0	
CW - 12	28.3	28.1	28.5	26.8	26.2	25.9	12.0	11.0	11.0	
CW - 20	29.6	29.6	30.6	28.1	27.8	28.2	9.0	9.0	9.0	
CW - 20	29.4	29.7	30.2	27.8	27.6	27.9	9.0	10.0	10.0	
CW - 20	29.4	29.8	30.6	27.6	27.8	27.9	9.0	10.0	10.0	
CW - 40	33.1	34.0	35.2	32.0	31.7	32.9	7.0	7.0	8.0	
CW - 40	33.4	33.9	35.2	31.9	31.8	32.7	7.0	8.0	8.0	
CW - 40	33.3	33.8	33.9	32.1	32.1	31.7	7.0	8.0	8.0	
CW - 60	36.9	37.1	38.6	35.7	35.6	36.5	4.0	6.0	5.0	
CW - 60	36.8	37.5	38.7	35.4	35.7	36.4	5.0	5.0	4.0	
CW - 60	36.7	37.5	38.4	35.6	35.5	35.5	6.0	4.0	5.0	
CW - 80	44.0	43.1	42.6	39.9	36.0	41.0	2.5	2.5	2.5	
CW - 80	43.9	43.0	42.4	39.8	39.7	41.3	2.5	2.5	2.5	
CW - 80	43.5	43.2	42.1	40.1	36.4	40.4	2.5	2.5	2.5	
CW - 88	46.9	45.8	47.6	43.8	43.6	45.4	2.5	2.0	2.5	
CW - 88	47.7	45.8	46.7	44.2	43.4	45.2	2.5	2.5	2.0	
CW - 88	47.1	44.9	46.4	43.8	43.6	44.9	2.5	2.5	2.0	
CW - 92	50.5	49.2	50.1	47.2	46.2	48.5	3.0	2.5	2.0	
CW - 92	50.2	48.9	50.4	46.3	45.6	49.1	3.0	2.5	2.5	
CW - 92	50.3	49.1	50.2	46.7	45.7	48.7	2.5	2.5	2.0	
CW - 96	55.6	53.2	54.5	53.6	49.9	49.9	1.5	1.5	2.5	
CW - 96	56.1	53.3	54.8	53.5	49.5	48.3	2.0	2.0	2.5	
CW - 96	55.5	453.2	54.4	52.9	48.8	48.7	2.0	2.5	2.5	

	DATA FROM AVERAGED MECHANICAL PROPERTIES VS. PERCENT COLD WORK - (ksi)									
	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
CONDITION										
HOT ROLLED	29.2	27.6	29.0	21.9	21.0	22.5	21.3	20.3	18.0	
ANNEALED	24.8	24.7	24.3	12.6	12.9	12.7	25.0	28.0	26.7	
CW - 12	28.2	28.0	28.6	26.6	26.1	26.1	10.7	11.0	11.0	
CW - 20	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7	
CW - 40	33.3	33.9	34.8	32.0	31.9	32.4	7.0	7.7	8.0	
CW - 60	36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
CW - 80	43.8	43.1	42.4	39.9	37.4	40.9	2.5	2.5	2.5	
CW - 88	47.2	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2	
CW - 92	50.3	49.1	50.2	46.7	45.8	48.8	2.8	2.5	2.2	
CW - 96	55.7	53.2	54.6	53.3	49.4	49.0	1.8	2.0	2.5	

DATA FROM 20% COLD WORK ANNEALING STUDIES - (ksi)									
ANNEALED AT 149°C (300 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7
2	31.8	32.1	32.6	27.3	26.5	26.6	11.0	10.5	10.0
4	32.4	32.2	32.9	27.0	26.1	26.5	11.0	11.0	11.0
8	33.4	33.2	33.6	26.9	23.1	26.3	14.0	14.0	14.0
16	33.7	33.5	34.1	27.2	26.5	26.6	14.0	14.0	14.0
32	34.2	33.5	34.6	27.4	26.4	26.6	14.0	14.0	14.0
ANNEALED AT 204 °C (400 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7
1	31.5	31.2	31.6	25.4	25.0	27.7	13.0	13.0	13.0
2	32.0	31.2	31.6	26.4	24.5	25.6	13.0	13.0	13.0
4	30.7	31.3	31.4	25.3	25.4	25.1	13.0	13.0	12.0
8	30.4	30.0	30.4	23.7	25.4	24.8	15.0	11.1	11.0
16	29.8	29.8	30.4	24.8	24.6	25.2	11.0	11.0	11.0
ANNEALED AT 246 °C (475 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7
1	28.2	28.1	28.5	22.7	21.9	22.1	16.0	15.5	15.5
2	28.1	27.6	27.7	21.7	21.3	20.9	15.0	15.0	15.0
4	27.7	27.2	27.7	21.4	20.1	21.6	15.0	15.0	15.0
8	27.6	27.3	27.7	21.7	21.3	21.6	17.0	17.0	15.0
16	27.3	27.0	26.9	21.0	20.6	21.0	17.0	17.0	17.0

DATA FROM 20% COLD WORK ANNEALING STUDIES - (ksi) (CONTINUED)										
ANNEALED AT 288 °C (550 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7	
0.5	27.2	27.1	27.1	21.4	20.7	21.0	17.0	17.0	17.0	
1	27.1	27.1	27.1	20.5	20.6	20.8	17.0	17.0	17.0	
2	27.5	26.7	26.8	20.6	19.9	20.0	17.0	17.0	17.0	
4	27.0	26.6	26.7	20.1	20.1	20.1	17.0	17.0	17.0	
8	26.8	26.5	26.0	19.7	19.8	20.1	17.0	17.0	17.0	
ANNEALED AT 316 °C (600 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7	
0.25	27.5	26.8	27.2	20.2	20.0	20.3	20.2	20.0	10.0	
0.5	27.4	27.1	26.5	20.0	19.5	19.7	18.0	18.0	18.0	
1	26.8	26.8	26.5	18.7	19.3	19.1	18.0	18.0	18.0	
2	26.9	25.9	26.0	18.5	17.0	17.6	18.0	18.0	18.0	
4	26.3	26.2	25.9	16.9	17.5	18.0	20.0	20.0	16.0	
ANNEALED AT 332°C (630 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	29.5	29.7	30.5	27.8	27.7	28.0	9.0	9.7	9.7	
0.125	27.3	26.9	27.1	19.6	19.6	19.8	16.0	18.0	20.0	
0.25	26.7	26.2	26.6	18.2	18.0	17.8	20.0	20.0	20.0	
0.5	26.1	25.6	25.9	15.9	16.1	16.2	20.0	20.0	20.0	
1	25.7	25.0	25.2	14.4	14.4	13.8	22.0	22.0	22.0	
2	24.9	24.7	24.9	12.2	13.0	13.4	22.0	22.0	22.0	

		DATA FROM 60% COLD WORK ANNEALING STUDIES - (ksi)									
ANNEALED AT 149°C (300 °F)											
TIME (HR)		UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0		36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
2		37.0	37.4	37.9	32.8	32.5	31.3	7.0	7.0	7.0	
4		37.8	37.5	38.2	32.5	31.2	32.9	7.0	7.0	10.0	
8		38.3	38.1	38.2	33.4	33.1	33.6	9.0	11.0	10.0	
16		38.6	39.3	38.2	33.8	34.4	33.5	12.0	12.0	10.0	
32		39.6	38.2	39.6	34.2	33.4	34.0	10.0	10.0	10.0	
ANNEALED AT 204 °C (400 °F)											
TIME (HR)		UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0		36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
1		32.0	31.8	32.8	27.6	26.8	28.5	13.0	15.0	15.0	
2		31.6	31.4	31.4	26.9	27.0	27.8	13.0	13.5	13.0	
4		30.9	30.8	31.0	26.5	26.4	27.1	13.5	13.5	9.0	
8		34.0	34.4	34.7	30.1	30.5	31.0	10.0	10.0	10.0	
16		33.1	33.4	34.1	29.2	29.3	30.4	10.0	10.0	10.0	
ANNEALED AT 246 °C (475 °F)											
TIME (HR)		UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0		36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
1		37.6	37.2	36.2	32.1	31.4	32.0	10.0	10.0	10.0	
2		36.2	36.4	36.8	31.2	31.7	32.1	10.0	10.0	10.0	
4		35.1	35.4	35.6	30.8	30.7	31.2	10.0	10.0	10.0	
8		30.7	30.5	30.5	25.4	26.2	26.6	12.0	12.0	12.0	
16		30.2	30.0	30.6	25.1	25.8	26.5	12.0	12.0	12.0	

DATA FROM 60% COLD WORK ANNEALING STUDIES - (ksi) (CONTINUED)										
ANNEALED AT 288 °C (550 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
0.5	29.1	28.7	29.0	21.1	21.6	22.0	15.0	17.0	15.0	
1	26.7	26.8	26.9	15.5	16.2	16.8	20.0	20.0	20.0	
2	25.8	25.0	25.1	12.3	11.6	12.0	21.0	25.0	26.0	
4	24.9	24.4	24.5	10.8	11.2	14.2	23.0	25.0	25.0	
8	25.4	24.3	24.1	11.6	11.0	10.6	25.0	25.0	25.0	
ANNEALED AT 316 °C (600 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
0.25	26.2	25.0	25.1	11.6	13.3	12.0	25.0	28.0	24.0	
0.5	25.8	24.9	25.0	10.8	10.7	11.1	25.0	25.0	25.0	
1	25.6	24.9	24.8	10.9	11.4	10.7	23.0	25.0	26.0	
2	25.8	25.0	25.0	10.8	10.6	10.9	27.5	26.5	24.5	
4	25.7	25.0	24.7	10.8	10.2	10.6	24.0	26.5	23.0	
ANNEALED AT 332°C (630 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	36.8	37.4	38.6	35.6	35.6	36.1	5.0	5.0	4.7	
0.125	25.7	25.0	25.0	11.1	10.5	10.8	25.0	25.0	26.0	
0.25	25.8	25.1	24.9	11.2	10.6	10.4	23.0	26.0	26.0	
0.5	25.6	25.2	25.0	10.6	10.4	10.5	24.0	27.7	28.0	
1	25.7	24.9	24.8	10.7	10.4	10.7	24.0	24.0	24.0	
2	25.7	24.9	25.0	11.2	10.2	10.9	24.0	25.0	25.0	

DATA FROM 88% COLD WORK ANNEALING STUDIES - (ksi)									
ANNEALED AT 149°C (300 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	47.7	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2
2	44.3	42.8	45.9	41.4	39.9	42.2	5.0	4.5	5.5
4	45.9	43.6	45.6	42.6	40.5	42.2	5.5	5.5	5.5
8	45.8	43.8	44.7	42.9	41.1	41.3	5.5	4.5	6.0
16	44.4	44.4	45.5	41.5	41.2	42.1	5.5	5.5	6.5
32	46.0	44.5	43.6	42.5	41.1	40.7	5.5	6.0	4.5
ANNEALED AT 204 °C (400 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	47.2	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2
1	42.4	41.4	42.1	38.2	38.4	38.6	5.5	5.5	5.5
2	40.3	39.7	41.2	36.7	36.9	38.5	6.0	5.5	6.5
4	39.2	37.9	39.0	35.7	35.3	36.8	6.5	5.0	3.5
8	37.3	36.8	37.9	33.9	34.3	35.6	6.5	6.0	5.5
16	36.3	35.7	36.8	33.2	33.7	34.8	7.0	6.5	6.0
ANNEALED AT 246 °C (475 °F)									
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°
0	47.2	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2
1	34.0	33.5	33.9	30.3	31.2	31.4	10.0	8.5	7.0
2	33.2	32.2	33.0	29.6	29.6	30.5	10.0	9.5	8.0
4	30.7	31.7	31.9	26.7	28.4	29.1	11.5	13.0	11.0
8	31.4	30.2	31.5	26.5	26.7	27.8	12.0	11.0	12.0
16	30.8	30.8	29.2	25.0	26.6	25.0	12.5	12.5	13.5

DATA FROM 88% COLD WORK ANNEALING STUDIES - (ksi) (CONTINUED)										
ANNEALED AT 288 °C (550 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	47.2	45.5	46.6	43.9	43.5	45.2	2.5	2.3	2.2	
0.5	24.6	26.4	27.5	13.8	14.7	16.3	7.0	20.5	20.5	
1	25.7	25.4	25.6	12.4	12.1	12.3	19.5	24.5	22.5	
2	26.1	23.9	24.9	12.1	11.3	12.1	22.5	20.5	20.5	
4	25.7	25.3	24.0	11.8	12.0	11.7	18.0	22.5	11.5	
8	25.9	25.2	25.0	12.1	11.7	11.8	19.0	25.0	21.5	
ANNEALED AT 316 °C (600 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	47.2	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2	
0.25	26.2	25.6	25.5	12.1	12.0	12.1	18.0	23.5	22.5	
0.5	26.2	25.7	25.5	12.2	12.3	11.9	23.0	24.5	22.0	
1	26.5	25.0	25.3	12.5	11.9	12.1	20.5	14.0	21.5	
2	26.1	25.4	25.5	12.1	11.5	13.3	19.0	21.5	22.0	
4	26.3	25.3	25.1	12.3	11.6	12.0	20.5	23.5	21.0	
ANNEALED AT 332°C (630 °F)										
TIME (HR)	UTS-0°	UTS-45°	UTS-90°	YS-0°	YS-45°	YS-90°	%ELONG-0°	%ELONG-45°	%ELONG-90°	
0	47.3	45.5	46.9	43.9	43.5	45.2	2.5	2.3	2.2	
0.125	26.3	25.8	25.8	12.2	11.9	12.3	20.0	23.0	20.5	
0.25	26.2	25.3	25.3	12.3	12.1	12.1	19.0	22.0	21.0	
0.5	26.5	25.6	25.6	12.4	11.8	12.2	19.5	17.0	21.5	
1	26.3	25.8	25.4	12.4	11.7	12.0	19.5	21.0	19.5	
2	26.3	25.9	25.6	12.1	12.2	12.1	19.0	21.5	21.5	

VITA

Tamara Kay McGill-Taylor was born in Knoxville, Tennessee on May 26, 1961. She graduated from Powell High School, June 1979, and immediately entered the college of engineering at the University of Tennessee, Knoxville.

During her sophomore year, she entered the Metallurgical Engineering Department. In June of 1983, she received a Bachelor of Science degree in Metallurgical Engineering.

In August of 1988, she returned to Knoxville to continue her studies in Metallurgical Engineering at the University of Tennessee, and received her Master of Science degree in December 1989.