

To the Graduate Council:

I am submitting herewith a thesis written by Mingli Yu entitled "The Effect of Heat Treatment and Cold Working on the Texture and Properties 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:

Edward S. Clark

W T Beck

Accepted for the Council:

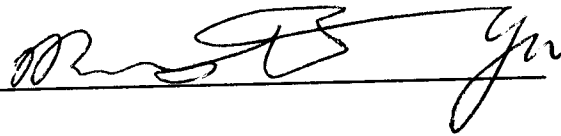
C W Minkel

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 

Date 7/26/91

The Effect of Heat Treatment and Cold Working on the Texture and Properties of Aluminum Alloy 3104

A Thesis

Presented for the
Master of Science
Degree

The University of Tennessee, Knoxville

Mingli Yu
August 1991

DEDICATION

To my wife and daughter

Huiyan and Lele

献给我妻子：慧艳，
女儿：乐乐。

ACKNOWLEDGEMENTS

My first appreciation is extended to my family-- my wife, Huiyan, and my parents, Mr. and Mrs. X. Yu, for their constant understanding, support, and encouragement.

I am extremely thankful to my major adviser, Dr. Charlie R. Brooks, for his great guidance and support throughout my fulfillment of the Master's degree in Metallurgical Engineering. I also appreciate Dr. W. T. Becker and Dr. E. S. Clark for serving as my committee members.

Great thanks are extended to T. K. McGill-Taylor, for her previous research to which I refer in my thesis here. I also appreciate very much what Mr. B. L. McGill has done for the scanning electron microscope work in the research.

Special thanks are extended to the staff of the Materials Science and Engineering Department for all their help during this project, especially the folks from the machine shop, Doug Fielden, Jack French, Ray Bellamy, Mike Rogers, and Mike Ensor, for their help; the secretaries of the department, Sandy Maples, Sue Turner, Phyllis Klindt, Kay David, and Pat Houser.

I would like also to thank Aileen Cagle, Randy Stooksbury, and Al Carter for their help and patience during the supply service of the project.

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

I am very grateful to Mr. Y. Xu and the people at the Department of Materials Science and Engineering, University of Pittsburgh, Pennsylvania, for assistance in obtaining the pole figures in the research. Acknowledgement is also extended to Dr. Lu and his colleagues at the Engineering Mechanics Department, University of Kentucky, Lexington, for the experiments of ultrasonic testing.

Special thanks are extended to Ravenswood Aluminum Corporation, Ravenswood, West Virginia for sponsoring this research project, and especially to Mr. Scott Goodrich for his constant understanding and support.

ABSTRACT

Five practices of annealing and cold rolling have been conducted in a 3104 Al alloy to investigate their influences on earing and the mechanical properties. Among these influences, slow heat-up rate (15°C/hr) to the annealing temperature of 332°C , which simulated batch annealing, and rapid heat-up rate (about 140°C/min.), which simulated strip annealing, both annealings proceeding 2 hours of time, were investigated, and some of the practices involved an intermediate cold rolling of 35% (CR35). The as-received (AR) condition was "hot" rolled state, and all final condition in the five practices was cold rolled 88% (CR88), so called "H19" state.

The tensile mechanical properties after the five practices were found out being independent of the direction of testing in the final CR88 state. The yield and tensile strengths were slightly lower for practices involving the intermediate CR35, however, these practices had markedly higher 45° earing. The higher heat-up rate gave a finer annealed grain size, but the heat-up rate had no effect (other steps of the processing being the same) on earing behavior.

Scanning electron microscopy and optical microscopy were used to examine the primary particle size, distribution, and density in the annealed state in all the five practices. However, no clear correlation with the earing occurrence.

X-ray diffraction, ultrasonic technique and pole figure were used to examine the texture in the five practices. The AR condition gave a complex texture (copper type) similar to that of all the final

CR88 conditions.

Results from both ultrasonic and pole figure techniques were consistent with those from the earing tests, practices involving the intermediate CR35 having higher earing and correspondently higher texture intensities. X-ray diffraction results were to reflect the major texture component variations during the different procedures in each of the five practices.

It appears that the finer microstructural features produced after the CR35 and intermediate annealing are responsible for the high earing observed. Therefore, further and deeper investigation should be focussed on this in other techniques such as transmission electron microscopy, etc.

TABLE OF CONTENTS

CHAPTER	PAGE
I. INTRODUCTION.....	1
II. LITERATURE REVIEW.....	5
II.1.Physical Metallurgy of 3xxx Al Alloys.....	5
II.1.1. Phases in 3xxx Al alloys.....	5
II.1.2. Casting	9
II.1.3. Homogenization.....	11
II.1.4. Annealing	19
II.1.4.1. Recovery.....	19
II.1.4.2. Recrystallization.....	19
II.1.4.3. The Mechanisms of Recrystallization.....	20
II.2.Mechanical Processing of 3xxx Al Alloys.....	27
II.2.1. Hot Rolling.....	27
II.2.2. Cold Rolling.....	28
II.2.3. Deep Drawing.....	32
II.3.Preferred Orientation and Texture in 3xxx Al Alloys.....	34
II.3.1. Rolling Texture.....	35
II.3.1.1. The Mechanisms of Rolling Texture Formation.....	39
II.3.1.2. Cold Rolling Texture of 3004 Al alloy.....	43
II.3.1.3. Hot Rolling Texture of 3004 Al Alloy.....	44
II.3.2. Annealing Texture.....	47
III. EXPERIMENTAL PROCEDURE.....	52
III.1.Material for the Experiment.....	52
III.2.Cold Rolling and Annealing.....	52
III.3.Mechanical Testing.....	58
III.3.1. Tensile Testing.....	58
III.3.2. Earing Cup Testing.....	58

CHAPTER

PAGE

III.4. Optical and SEM Metallography.....	5 9
III.5. X-Ray Diffraction Intensity Measurements.....	6 1
III.6. Ultrasonic Testing.....	6 3
III.7. Pole.Figure.....	6 4
IV. RESULTS AND DISCUSSION.....	6 7
IV.1. Mechanical Properties.....	6 7
IV.2. Microstructure and Particle Examinations.....	7 2
IV.3. Texture Detection and Analysis.....	9 3
IV.4. Closure.....	1 1 2
V. CONCLUSIONS AND PROJECTIONS.....	1 1 6
V.1. Conclusions.....	1 1 6
V.2. Projections.....	1 1 8
REFERENCES.....	1 2 0
APPENDICES.....	1 2 9
Appendix 1.....	1 3 0
Appendix 2.....	1 3 3
Appendix 3.....	1 3 4
Appendix 4.....	1 3 5
VITA.....	1 3 9

LIST OF TABLES

TABLE	PAGE
1. Processing sequence of each of the five practices studied.....	4
2. Major components of texture [45].....	3 8
3. Cube texture strengths in as-rolled material [32].....	4 6
4. Measurements of texture (ODFs) for various alloy treatments [33].....	4 8
5. Composition of 3104 Al alloy (compared with the nominal composition of 3104 and 3004).....	5 3
6. Particle percentage by IPM method.....	8 9
7. EDS microanalysis of primary particles (AR condition).....	9 0
8. Primary particle density for the annealed conditions of the five practices.....	9 1
9. X-ray diffraction results of Prac. 3-1 in multi-layer etch off method.....	1 0 1
10. Maximum intensities in {111} pole figures from the five final CR88 states.....	1 1 1
11. Ultrasonic test results from final CR88% condition of all 5 practices.....	1 1 4

LIST OF FIGURES

FIGURE	PAGE
1. Phase diagram of aluminum-manganese system [2].....	6
2. Phase diagram of aluminum-magnesium system [3].....	7
3. Solidus of the Al-Fe-Si Ternary phase diagram [5].....	8
4. A scheniac diagram of the DC casting method [8].....	1 1
5. Microstructure of 3003 alloy as-cast ingot showing the distribution of predominantly $Al_6(Mn, Fe)$ (light) and $Al_{12}(Fe, Mn)_3Si$ (dark) at dendrite interstices. 10% phosphoric acid, 455x [13].....	1 2
6. Microstructure of 3003 alloy homogenized ingot showing the transformation to the predominantly $Al_{12}(Fe, Mn)_3Si$ 10% phosphoric acid, 455x [13].....	1 2
7. Microstructure of 3003 alloy homogenized ingot, similar to Fig. 6 but etched to reveal dispersoid precipitates in the primary aluminum dendrite matrix. 0.5% hydrofluoric acid, 455x [13].....	1 3
8. TTT Diagram for formation of α -dispersoid for alloy 3004: (a) complete transformation for alloy S with excess (0.35) Si and (b) 50% transformation C-curves for basic (0.20%) Si alloy B, high (0.35%) Si alloy S and high (1.60) Mg alloy M [11].....	1 5
9. Effect of Homogenization Temperature and Heating Rate during Annealing on Post-Annealing Earing for 3004 and 1100 Aluminum Alloy [16].....	1 7

FIGURE

10.	Effect of homogenization temperature on annealed and cold rolled alloy 3004 [18].....	1 8
11.	Grain boundary between a growing crystal (I) and the rest of the specimen (II) [22].....	2 2
12.	Section of a binary equilibrium diagram with limited solid solubility [22].....	2 5
13.	Interaction between grain boundary and spherical inclusion [22].....	2 6
14.	Effect of finish hot rolling temperature on earing level of annealed (BA) hot bands, with homogenization S* and typical iron content For Alloy 3004 [33].....	2 9
15.	Substructure evolution in rolled 3004 [34].....	3 1
16.	Drawn aluminum cups (57% reduction) showing (a) 90° earing; (b) 45 ° earing; (c) mixed 90° and 45° earing; and (d) no earing; with respect to rolling direction, RD, shown in the arrow [13].....	3 3
17.	(111) pole figure of AA3004 90% reduced at room temperature [43].....	3 6
18.	Effect of temperature on the $\phi=45^\circ$ section of ODF in 3004 alloy [43]. (a) Rolling at room temperature, (b) rolling at 520 °C.....	3 7
19.	The deformation of a cube-shaped crystal using the different stress systems [50].....	4 1

FIGURE

20.	Calculated pole figures for (III) + (IV) + random stresses, 80% reduction, and experimental copper type pole figures, 94% reduction, a) {200}, b) {111} pole figures [50].	4 2
21.	Ratio of average of 12 intensities about $(\bar{1} \ 1 \ 0)[1 \ \bar{1} \ 2]$ over $(\bar{1} \ 1 \ 2)[1 \ \bar{1} \ 1]$ on $\phi = 45^\circ$ section of ODF against reduction [43].	4 5
22.	This diagram illustrates the general trend in the variation of the strength of the cube texture component with alloy content for 3xxx alloys [24].	5 0
23.	The recrystallized grain size and the strength of the cube texture component as a function of the parameters given [24].	5 1
24.	Schematic plot of slow heat-up rate anneal (BA). WQ stands for "water quenched".	5 5
25.	Schematic plot of fast heat-up rate annealing (SA). WQ stands for "water quenched".	5 6
26.	A recording of the fast heatup rate annealing.	5 7
27.	Metallurgical samples cut off from the tensile bars [4].	6 0
28.	X-ray samples cut off from the tensile bars.	6 2
29.	X-ray samples cut off from the earing cup bottom.	6 2
30.	Shulz reflection method for pole figure formation [59].	6 5
31.	The tensile mechanical properties at each stage in each processing practice.	6 8

FIGURE

32.	The effect of processing practice on the tensile mechanical properties for the final 88% cold worked condition.....	7 0
33.	The effect of processing practice on the degree of earing (45°) for the final 88% cold worked condition.....	7 1
34.	Grain structure of the AR condition. 200X.....	7 3
35.	Grain structure of Prac. 1. 200X	7 4
36.	Grain structure of Prac. 2. 200X	7 5
37.	Grain structure of Prac. 3. 200X.....	7 6
38.	Grain structure of Prac. 4. 200X	7 7
39.	Grain structure of Prac. 5. 200X.....	7 9
40.	Optical micrograph, showing primary particles.....	8 0
41.	SEM micrographs (back - scattered electrons) of the material in the AR hot rolled condition. The specimen was unetched.....	8 4
42.	SEM micrographs of the material in Pracs. 1 and 2 prior to the final CR88% state.....	8 5
43.	SEM micrographs of the material in Pracs. 3 and 4 prior to the final CR88% state.....	8 6
44.	SEM micrographs of the material in Prac. 5 prior to the final CR88% state.....	8 7
45.	Primary particle percentage in prac. 3 before the final CR88% state by IPM.....	8 8

FIGURE

46.	{200}, {220} plane x-ray diffraction for Pracs. 1 and 2.....	9 4
47.	{200}, {220} plane x-ray diffraction for Pracs. 3 and 4.....	9 5
48.	{200}, {220} plane x-ray diffraction for Prac. 5.....	9 6
49.	{422} plane x-ray diffraction ratio after CR88 of all practices, AR condition is plotted on the left.....	9 7
50.	{200}, {220}, and {422} plane x-ray diffraction for all the final CR88% condition.....	9 8
51.	{111} pole figure for AR condition.....	1 0 3
52.	{111} pole figure for Prac. 1 in final CR88% condition.....	1 0 4
53.	{111} pole figure for Prac. 2 in final CR88% condition.....	1 0 5
54.	{111} pole figure for Prac. 3 in final CR88% condition.....	1 0 6
55.	{111} pole figure for Prac. 4 in final CR88% condition.....	1 0 7
56.	{111} pole figure for Prac. 5 in final CR88% condition.....	1 0 8
57.	{200} pole figure for Prac. 2 in final CR88% condition, comparing with Fig. 52.....	1 0 9
58.	The relation between the mean earing percentage and the ratio of ($R_0, 90-R_{45}$)/ R_a . [37].....	1 1 3

INTRODUCTION

Due to their light weight and drawability, aluminum alloys are used for the can stock industry. They have excellent resistance against corrosive elements in the atmosphere, water (including salt water), oils and many chemicals [1].

One of the typical aluminum alloys for packaging is the 3xxx series. 3xxx series Al alloys are based on 1xxx series, which are industrially pure Al, with added Mn. Usually the Mn content is about 1 wt%, giving the alloys about 20% more strength than 1xxx alloys. 3003, the first one in use about 50 years ago, had 1% Mn in it. Later, 3004, which had about 1% Mg in it soon substituted 3003 -- are widely used as general-purpose alloys for moderate strength applications requiring good workability. These applications include beverage cans, cooking utensils, heat exchangers, storage tanks, awnings, furniture, highway signs, roofing, siding and other architectural applications.

There are some new specifications of 3xxx in application. Al alloy 3104, only with higher Mg content compared with 3004, and closer control of some impurities, so with a little lower strength than 3004, better ductility (comparing at the same H19 state), manufactured by Ravenswood Aluminum Corporation, is one of them.

During beverage can formation, major operations such as hot rolling, cold rolling, and deep drawing, may develop or alter texture and therefore may cause undesirable earing.

The purpose of this research project is to minimize the earing occurrence, which is believed to have a great deal to do with texture during the can manufacture hence to lower down the consumption of energy, time, and consequently expenses.

In order to achieve the purpose, scientists and metallurgists have been struggling for decades by trying close control of all major metallurgical factors, such as composition, casting method, homogenization, hot rolling, annealing, cold rolling, and so on. A great deal of efforts are focused on the mechanisms of the texture formation, yet the results are still beyond satisfaction.

Texture or preferred orientation occurred during the deformation is called deformation texture. It is considered that this texture is due to the tendency of the grains in a polycrystalline aggregate to rotate during plastic deformation. Each grain undergoes slip and rotation in a complex way that is determined by the imposed stresses and by the slip and rotation of adjoining grains; the result is a preferred, nonrandom orientation.

When the cold-worked metal, with a deformation texture, is recrystallized by annealing, the new grain structure usually has a preferred orientation too, often different from that of the cold-worked material. This is called a recrystallization texture or annealing texture. It is due to the influence which the texture of the cold-worked matrix has on the nucleation and/or growth of the new grains in that matrix.

It is known that the type and distribution of dispersoids, which are controlled by heat treatment (homogenization and annealing),

affect the preferred orientation or texture.

The research work being proposed here deals with an examination of these factors that affect the mechanical property directionality, texture, and hence earing, in the can stock Al alloy 3104. Specifically, the effects of heat-up rate to the annealing temperature, of the amount of prior cold work and the annealing time and temperature, and of the intermediate cold rolling and intermediate annealing on mechanical properties, texture or earing are to be determined for this alloy. This information is to be used to develop an optimum processing sequence to minimize earing in this material.

Therefore, five practices were proposed in this research. The intermediate cold rolling reduction was 35%, combining with different heat-up rate to annealing temperature of 332 °C (630 °F), and final cold rolling reduction was 88%, which is the most common H19 strain hardened state. These can be seen in Table 1.

It should be noted that one simply can not address the texture problem in just one or two texture components in 3xxx alloys, and the efforts to understand this are too enormous to mention here.

Table 1 Processing sequence of each of the five practices studied.

Practice 1	Hot rolled (HR) + batch annealed* (BA) + cold rolled 88%(CR88)
Practice 2	Hot rolled + strip annealed* (SA) + cold rolled 88%
Practice 3	Hot rolled + cold rolled 35% (CR35) + batch annealed* + cold rolled 88%
Practice 4	Hot rolled + cold rolled 35% + strip annealed* + cold rolled 88%
Practice 5	Hot rolled + batch annealed* + cold rolled 35% + batch annealed* + cold rolled 88%

Note: This initial 'hot' rolled condition was the as-received condition of 0.503 cm (0.198 in.) thick plate.

* Samples annealed for 120 min. at 332°C (630°F). "Batch" and "strip" anneal will be discussed later.

LITERATURE REVIEW

For 3xxx Aluminum alloy can stock products, the following major thermomechanical processing is involved:

- * CASTING
- * HOMOGENIZATION
- * HOT ROLLING
- * ANNEALING(RECOVERY AND RECRYSTALLIZATION)
- * COLD ROLLING
- * DEEP DRAWING.

During these steps, especially casting and homogenization, some important constituent formation has to be examined in terms of some phase diagrams, such as Al-Mn, Al-Mg, and Al-Fe-Si, These diagrams can be seen in Fig. 1 [2], phase diagram of aluminum-manganese system, Fig. 2 [3], phase diagram of aluminum-magnesium system, and Fig. 3 [5], solidus of the Al-Fe-Si ternary phase diagram.

II.1. Physical Metallurgy of 3xxx Al alloys

II.1.1. Phases in 3xxx Al alloys

From Fig. 1, β , which is Al_6Mn and orthohombic, is the primary particle formed during casting in 3xxx aluminum alloys. Due to Fe and other element additions to Al-Mn alloy, the maximum solubility of manganese in aluminum is lowered from 1.82 wt% to 1.25 wt% [4]. Thus a large portion of Mn exists in 3xxx series as particle formers.

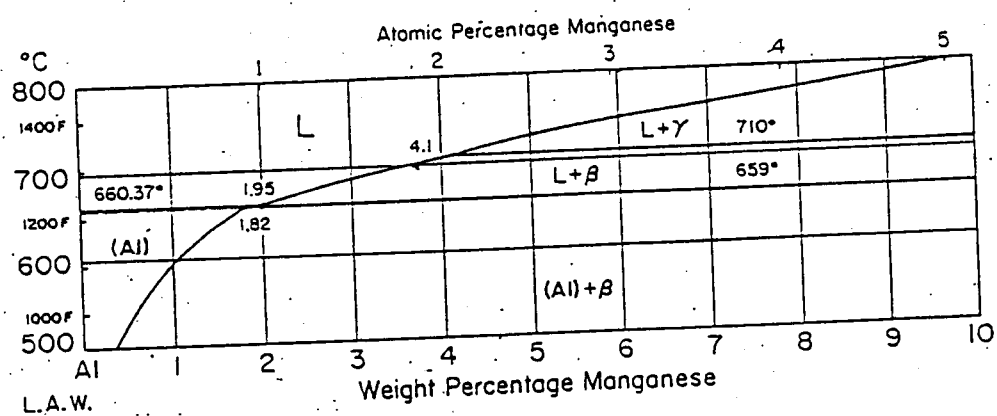
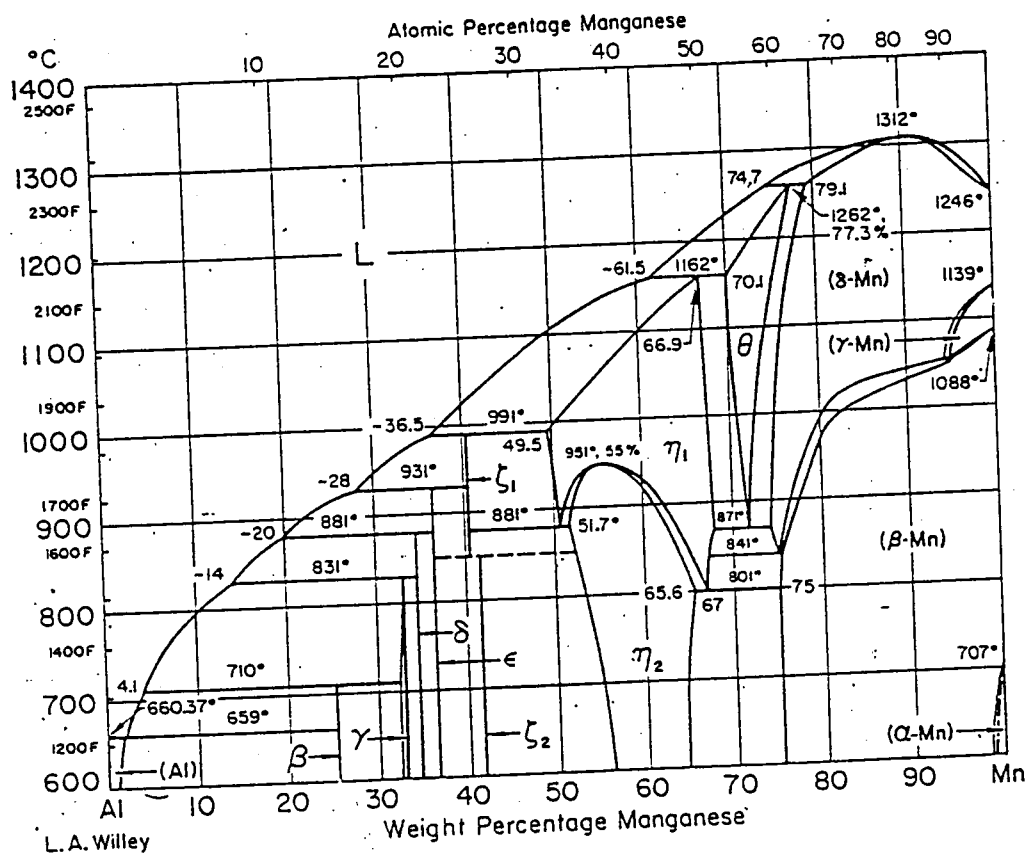


Fig. 1 Phase diagram of aluminum-manganese system [2].

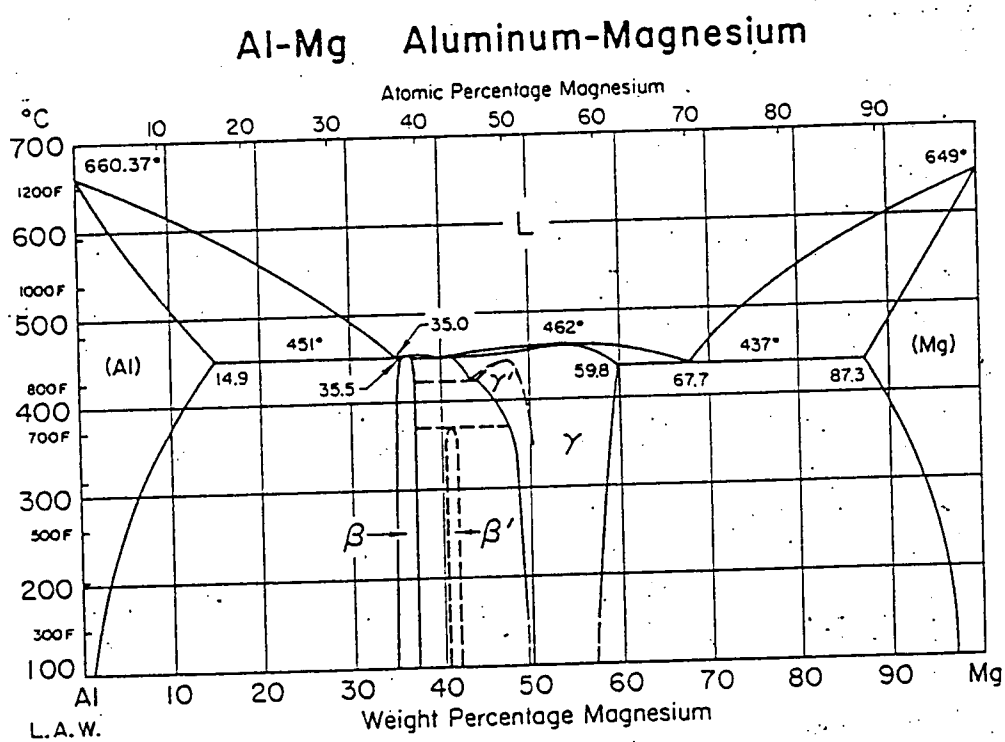


Fig. 2 Phase diagram of aluminum-magnesium system [3].

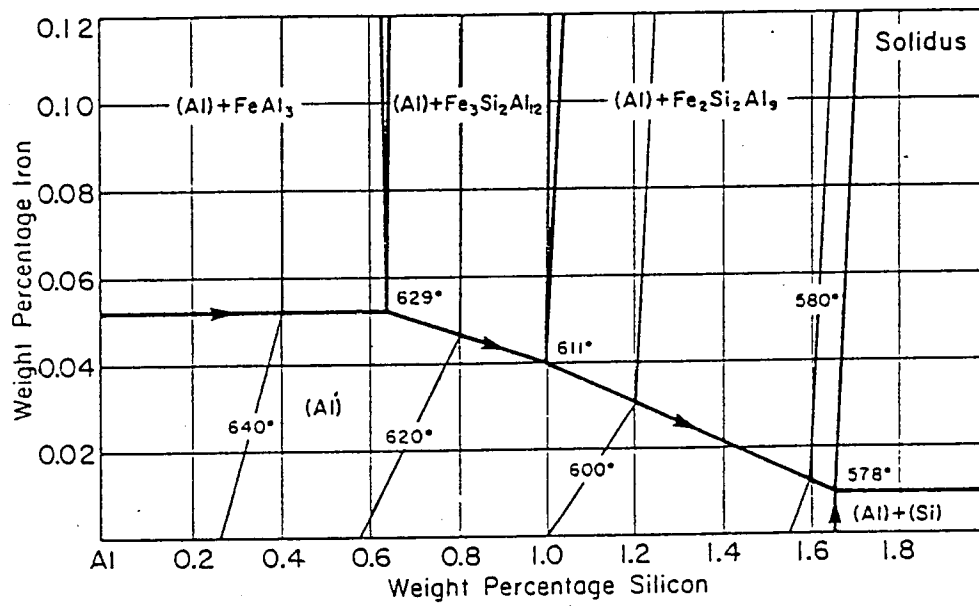


Fig. 3 Solidus of the Al-Fe-Si ternary phase diagram [5].

In Fig. 2, when the Mg content is about 1 wt%, no Mg compounds exist. Mg is in solid solution in the Al matrix, which is also the case in 3xxx series alloys.

From Fig. 3, a few kinds of binary and ternary phases occur, such as Al_3Fe , $\text{Al}_9\text{Si}_2\text{Fe}_2$, $\text{Al}_{12}\text{Si}_2\text{Fe}_3$, and Al_3SiFe . Sometimes the last three are referred to as α -type primary particles [5]. It should be pointed out that α -type primary particles may have different subscripts and have a variable composition, which we will encounter later in the thesis.

II.1.2. Casting

3004 Aluminum alloy has a melting range of $629\text{-}654^\circ\text{C}$ ($1165\text{-}1210^\circ\text{F}$) [6]. 3104 Aluminum alloy begins to melt at about 623°C (1170°F) [7].

The most commonly used way of casting 3xxx aluminum alloys today is by the direct-chill (DC) semi-continuous method Fig. 4 [8]. Sometimes strip casting is also very common [9]. The major difference between DC casting and strip casting is the cooling rate (strip casting is about 1000 times faster than DC casting [10]), and because of this, strip casting gives a higher solid solution supersaturation [9, 10].

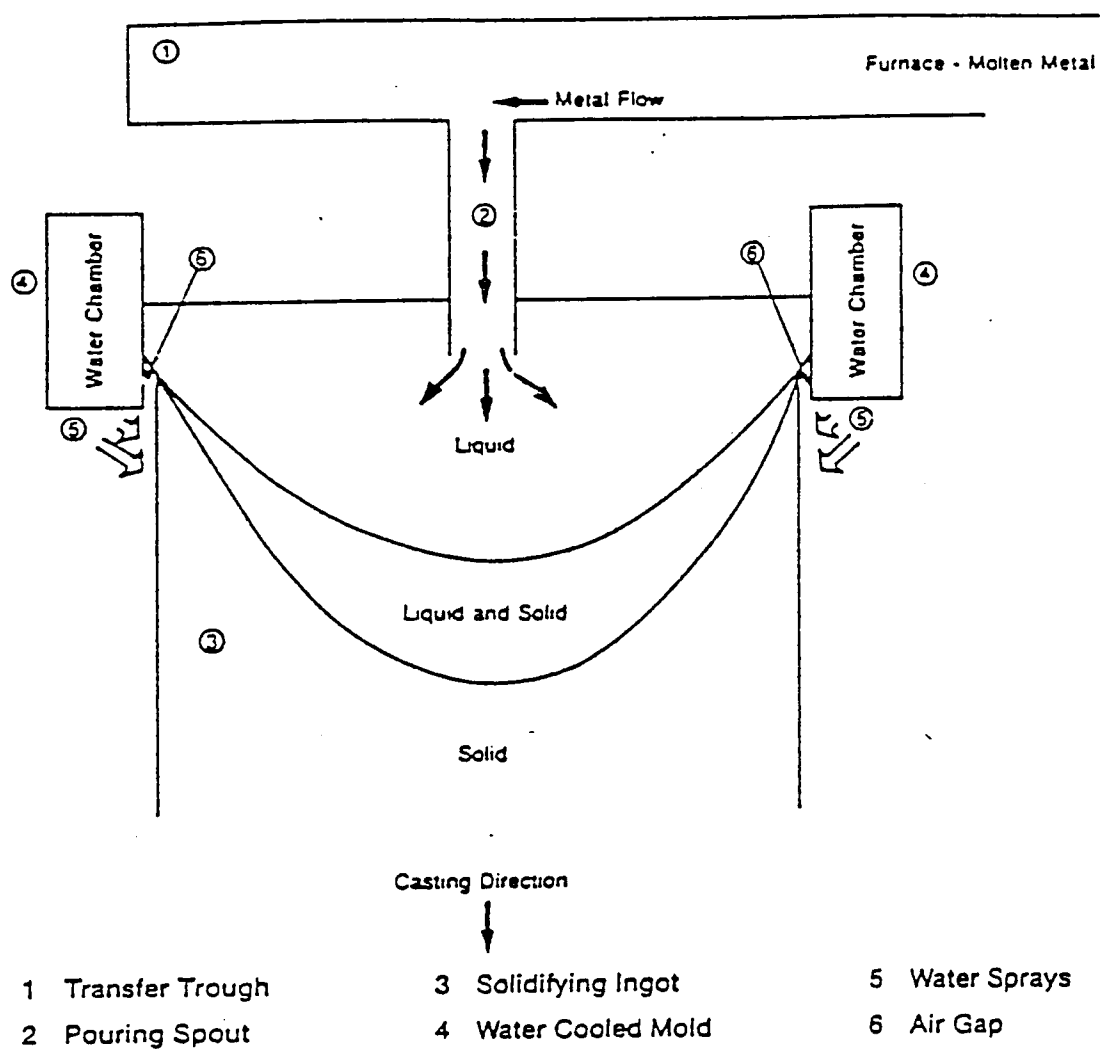


Fig. 4 A schematic diagram of the DC casting method [8].

During casting, due to the extremely fast cooling rate, only a small portion of Mn precipitates as primary particles and the majority of it is retained in the solid solution. The primary particles are $\text{Al}_6(\text{Mn,Fe})$, Al_6Mn , α -type $\{\alpha\text{-Al}_3\text{Fe}_2\text{Si}$, $\alpha'\text{-Al}_{20}\text{Fe}_5\text{Si}_2$ and $\alpha\text{-Al}_{12}(\text{Mn,Fe})_3\text{Si}\}$ [9]. Some Mn_2Si particles are also formed [7, 11]. In later homogenization, Mn_2Si will be dissociated into α -type dispersoid preferentially as the preheat temperature of ingots is reached above 428°C (800°F) [7], and a part of $\text{Al}_6(\text{Mn,Fe})$ will transform to α -type [7, 11]. There are evidences that coarser and denser primary dispersoids are connected with higher 45° earing percentage in final deep drawing [12].

II.1.3. Homogenization

Homogenization, sometimes also called soaking or preheating, is the process of decreasing the microsegregation by heating the casting ingots to promote the diffusion of solute atoms within the grains.

It is understood that during homogenization the primary phase $\text{Al}_6(\text{Mn,Fe})$, which is orthorhombic, will transform into α -type $\{\alpha\text{-Al}_3\text{Fe}_2\text{Si}$, $\alpha'\text{-Al}_{20}\text{Fe}_5\text{Si}_2$ and $\alpha\text{-Al}_{12}(\text{Mn,Fe})_3\text{Si}\}$, which are primitive-cubic. Mn supersaturated in the cored solid solution of the primary dendrites during the casting will subsequently precipitate as a dispersoid [13], shown in Figs. 5, 6, and 7. A few alloys in this group also contain Mg which, because of its affinity for silicon, tends to shift the phase proportioning toward $\text{Al}_6(\text{Mn,Fe})$. 3104 alloy is just this kind of alloy. Studies [12] showed that transformation of eutectic constituents from $\text{Al}_6(\text{Mn,Fe})$ to α -type would proceed in 3104 Al

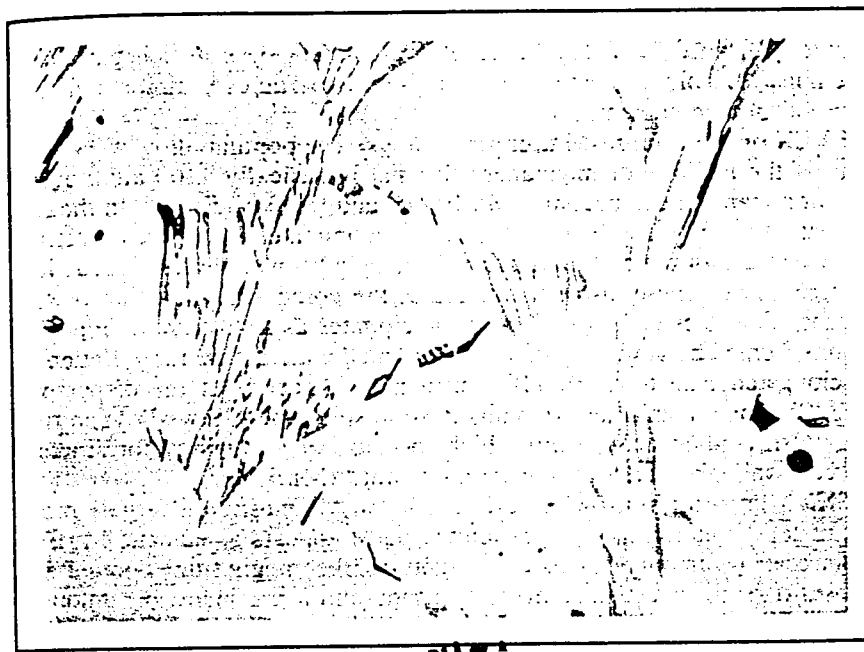


Fig. 5 Microstructure of 3003 Alloy as-cast ingot showing the distribution of predominantly $\text{Al}_6(\text{Mn,Fe})$ (light) and $\text{Al}_{12}(\text{Fe,Mn})_3\text{Si}$ (dark) at dendrite interstices. 10% phosphoric acid, 455x [13].

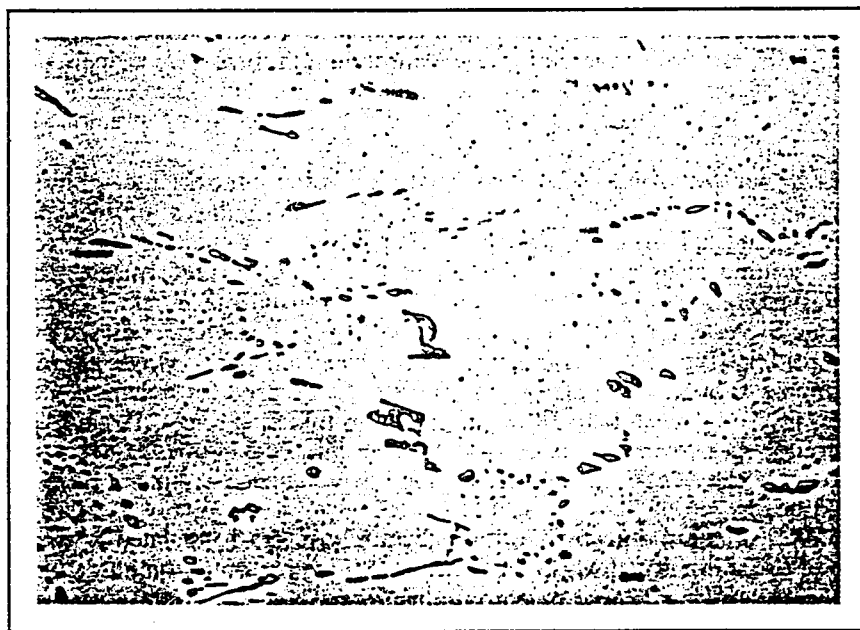


Fig. 6 Microstructure of 3003 Alloy homogenized ingot showing the transformation to the predominantly $\text{Al}_{12}(\text{Fe,Mn})_3\text{Si}$. 10% phosphoric acid, 455x [13].

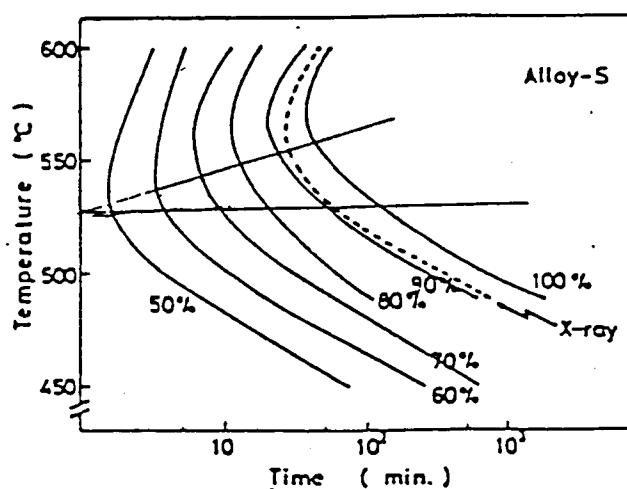


Fig. 7 Microstructure of 3003 Alloy homogenized ingot, similar to Fig. 6 but etched to reveal dispersoid precipitates (shown by the arrows) in the primary aluminum dendrite matrix. 0.5% hydrofluoric acid, 455x [13].

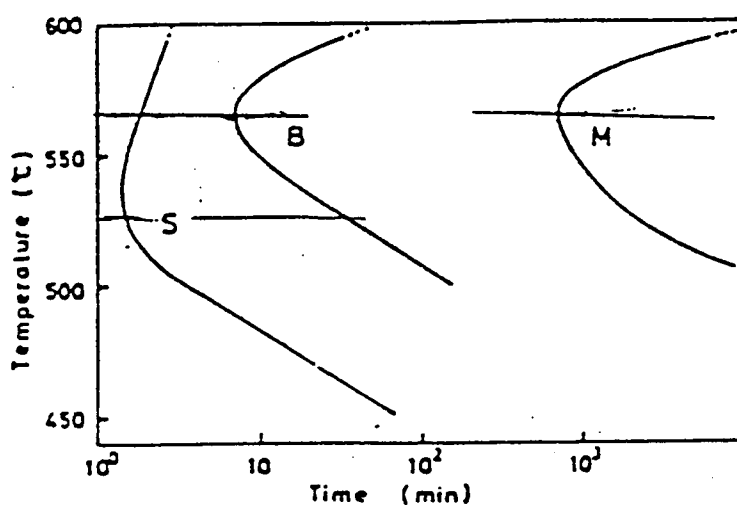
Both homogenization temperature and Si content affect the kinetics of α -type formation [11]. Fig. 8 shows a TTT curve for α -dispersoid formation of alloy 3004.

Studies [14] showed that higher homogenization temperature could increase the average sizes of both the primary particles and the smaller secondary particles and favor α - $\text{Al}_{15}(\text{Mn,Fe})_3\text{Si}_2$ formation. Increasing Si content promoted the formation of α - $\text{Al}_{15}(\text{Mn,Fe})_3\text{Si}_2$, consistent with the TTT curves mentioned above. These results were also verified in Chen and Morris' work [15]. They pointed out that in the strip cast 3004 alloy, when the homogenization temperature was 260°C , the secondary precipitate particles were $0.005\text{-}0.01\ \mu\text{m}$ diameter and $0.1\text{-}0.2\ \mu\text{m}$ long and needlelike, while at 450°C they were $0.02\text{-}0.03\ \mu\text{m}$ thick and $0.1\text{-}0.2\ \mu\text{m}$ long and platelike.

All the primary and secondary particle characteristics (the particle size, particle density, particle fraction, and particle distribution) may influence the earing tendency in terms of a process of competition between cube-oriented recrystallized grains and other oriented grains [16]. For example, rather large and coarsely dispersed primary particles precipitated during annealing following the homogenization, promoted the cube texture formation (cube texture will be discussed in Chapt.II.3.2. later); however, fine Fe and Si rich phases precipitating at the recrystallization front, hindered the development of cube texture [17].



(a)



(b)

Fig. 8 TTT diagram for formation of α -dispersoid for alloy 3004: (a) transformation for alloy S with excess (0.35%) Si and (b) 50% transformation C-curves for basic (0.20%) Si alloy B, high (0.35%) Si alloy S and high (1.60%) Mg alloy M [11].

Here is an example of processing 3004 alloy [11]:

The 3004 alloy was cast into 50 cm thick ingot
Homogenized, heating at 40 °C/s, and cooled at 20 °C/s
Hot rolled at 460 °C entry and 260 °C exit to 0.42 cm thickness
Cold rolled to 0.1 cm thickness
Annealed: (a) box type, heating rate 25 °C/hr to 350 °C, holding
2 hrs; (b) continuous annealing (CA) at 25 °C/s to 500 °C, holding a
few seconds

The results of the homogenization temperature are seen in Fig. 9.

Kitao [18] independently obtained similar results for 3004 alloy, shown in Fig. 10. Here the cast ingot underwent a batch anneal (or box-type anneal) and then was cold rolled. Lowest 45° earing (earring will be covered in Chapt. II.2.3.) corresponds to the 535°C homogenization. At lower annealing temperatures, the recrystallization is incomplete, and therefore the cube texture strength (Chapt. II.3.2.) is smaller, that is, the 45° earing strength is higher (Chapt. II.2.3.), especially at temperatures below the optimum homogenization temperature.

Fine dispersed particles prior to rolling were considered [19] to have a tendency to increase the pure Cu type texture component* (Chapt. II.3.1.) by decreasing the brass type component. When a volume fraction of particles larger than 1 μm went over about 2%, development of the usual deformation textures was hindered and textures tended to become random [20].

* The author [19] used {146}<211> as the pure Cu texture component.

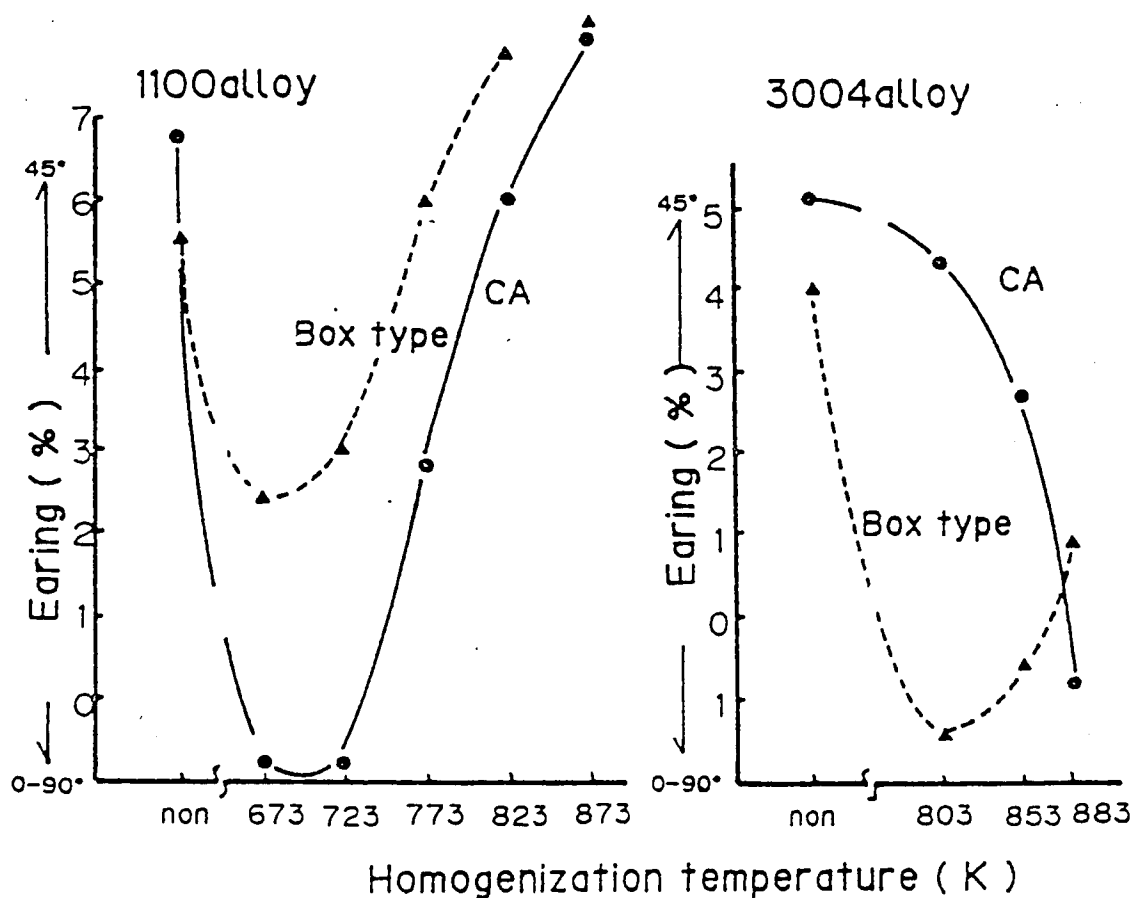


Fig. 9 Effect of homogenization temperature and heating rate during annealing on post-annealing earing for 3004 and 1100 aluminum alloy [16]. The optimum homogenization hold is about 530 °C (803 K) for box-type anneal and about 610 °C (883 K) for the CA, which corresponds approximately to the TTT curve nose of 3004 alloy in Fig. 8 [11]. These are the temperatures for most rapid precipitation of Fe and Mn from the cold rolled supersaturated matrix.

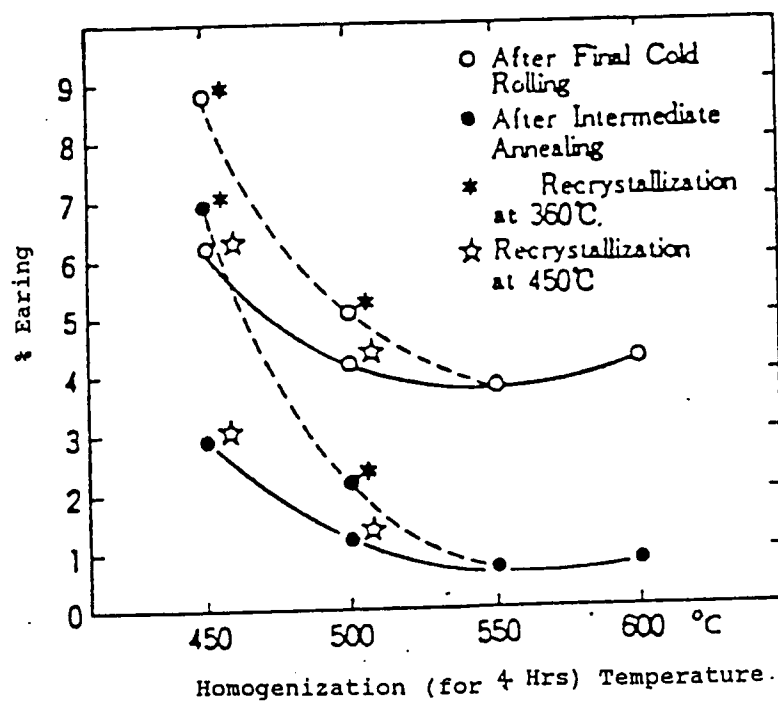


Fig. 10 Effect of homogenization temperature on annealed and cold rolled alloy 3004 [18].

II.1.4. Annealing

The dislocation structure after being plastically deformed (either hot or cold, but mainly cold) of 3xxx Al alloys is mechanically stable, but thermodynamically not stable. Therefore, the as-deformed structure always tends to lower its Gibbs free energy by the reduction and rearrangement of the lattice defects to revert to the annealed state which is thermodynamically stable. The whole process is often referred to as annealing. Through this process, there are a series of phenomena occurring for the material to approach a state of equilibrium, fundamentally the following: recovery, recrystallization, and grain growth.

II. 1.4.1. Recovery

Recovery is often defined as the restoration of some of the physical properties of cold worked metal without evident changes of microstructure. The structural changes involve essentially subgrain formation and enlargement.

In general, the subgrains in rolled aluminum are much less elongated in the rolling direction than are the grains, indicating that considerable sub-boundary movement toward a more equiaxed shape has occurred [21].

II. 1.4.2. Recrystallization

Recrystallization (here meaning primary recrystallization) is characterized by the formation of a new set of strain-free grains and the growth of these into the surrounding deformed material. The

new structure contains few dislocations within the grains. Recrystallization is easily examined by metallographic methods and is clearly seen by a decrease in hardness or strength and an increase in ductility. Besides, almost all influences of strain hardening are gone. It occurs with longer times or higher temperatures than do the recovery effects described in the preceding section, but of course some overlapping of the two processes is unavoidable.

In the present research, the nominal composition is fixed, 3104 alloy, but the thermal processing will affect the composition in the solution, and the dispersoids. By altering the temperature, time, and especially the amount of prior deformation, the influence on recrystallization, and furthermore, texture characteristics, are affected.

Since recrystallization dominates the annealing process, especially the texture properties, the mechanisms of recrystallization are focused on in my thesis here.

II.1.4.3. The Mechanisms of Recrystallization

The mechanisms of nucleation during recrystallization has been summarized by Doherty [21], and the following description is based on his treatment.

During the recrystallization, the recrystallized grain boundaries will move into the unrecrystallized grain boundaries as the Gibbs

free energy of the system is lowered. This is illustrated in Fig. 11 [22], where

F -- the cross sectional area in a rectangular body;

L -- the total length of the rectangular body;

g_I -- free energy per unit volume in grain I, which is the recrystallized grain;

g_{II} -- free energy per unit volume in grain II, which is still the cold worked grain;

G_{tot} -- total free energy of this system.

$$G_{tot} = F(xg_I + (L-x)g_{II}) \quad \text{eq. 1}$$

$$dG_{tot}/dx = -F(g_{II} - g_I) < 0 \quad \text{eq. 2(a)}$$

$$P_d = -(1/F)dG_{tot}/dx = g_{II} - g_I > 0 \quad \text{eq. 2(b)}$$

The recrystallization driving force p_d is $g_{II} - g_I$, and must be greater than zero for the grain boundary to move into the unrecrystallized (cold worked) grain. It is the resultant driving force and should be also expressed as the following in terms of the mechanics on the grain boundary movement:

$$P_d = \sum P_i \quad \text{eq. 3}$$

P_i -- individual force term exerted on the grain boundary (between grain I and grain II) by individual energy change term.

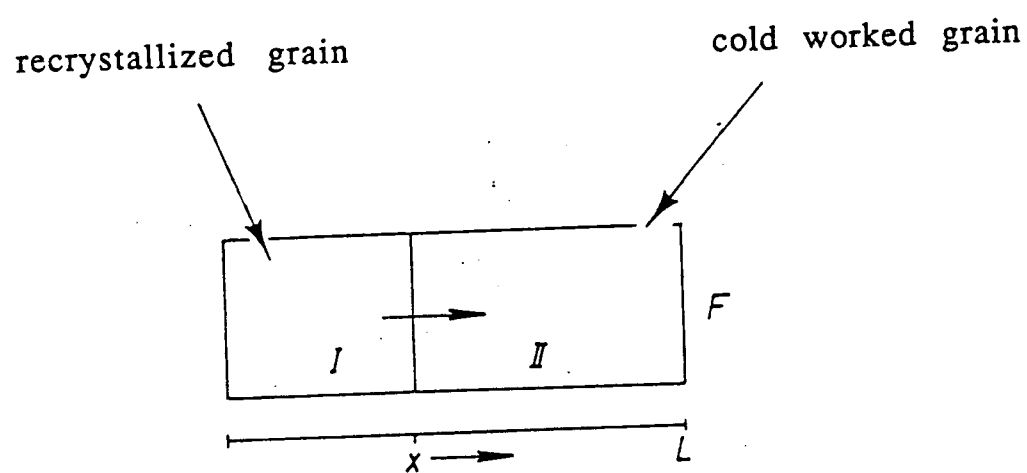


Fig. 11 Grain boundary between a growing crystal (I) and the rest of the specimen (II) [22].

$$P_d = \sum P_i \quad \text{eq. 3}$$

P_i --individual force term exerted on the grain boundary (between grain I and grain II) by individual energy change term.

More specifically, if P_i is positive, then it can be defined as driving force; if P_i is negative, then it can be defined as dragging force.

The driving force is caused by certain energy increase terms, seen in eq. 2(b), (we can also say "energy decrease term", seen in eq. 2(a)), such as the stored energy from cold work, P_1 ; grain boundary energy, P_2 ; molar free energy difference between supersaturated and equilibrium solid solution, P_3 ; and etc [22]. These are given by:

$$P_1 = Gb^2(\rho_{II} - \rho_I) \quad \text{eq. 4}$$

G -- the shear modulus of the material,

b -- the Burger's vector length,

ρ_{II}, ρ_I -- the dislocation densities in grain II and I respectively. Normally, $\rho_{II} \gg \rho_I$, therefore, $P_1 \approx Gb^2\rho_{II}$.

$$P_2 = (6D^2\gamma_B)/(2D^3) = 3\gamma_B/D \quad \text{eq. 5}$$

γ_B -- specific grain boundary energy,

D -- cube edge of grain I, assuming the average grain as cube shape.

$$P_3 = R(T_1 - T_0)c_0 \ln c_0 \quad \text{eq. 6}$$

R -- the ideal gas constant,

T_1 , T_0 , c_1 , and c_0 -- seen in Fig. 12.

The dragging force is caused by certain energy decrease terms (eq. 2(b)), such as the dragging force associated with second phase particles (Zener Drag), P_4 .

$$\begin{aligned} P_4 &= Z d(\pi y^2 \gamma_B) / dx \\ &= -2 Z \gamma_B (\pi x) \end{aligned} \quad \text{eq. 7}$$

Z -- the number of second phase particles per cm^2 ,

γ_B -- same as above, specific grain boundary energy,

r , x , and y -- seen in Fig. 13.

Zener drag effect has been one of the major research concerns in the last a few decades [23-29], because it connects particles with the dislocation development, subgrain structure, nucleation sites, grain growth, and in turn, mechanical properties, and texture formation.

Humphreys et al. [29] specifically pointed out that closely spaced particles inhibited recrystallization, whereas large widely spaced particles promoted recrystallization, and the same tendencies could be found in nucleation and growth rates. If the particles were nondeformable during the deformation, large amount of dislocations would be generated at the particles. For particles of diameter less

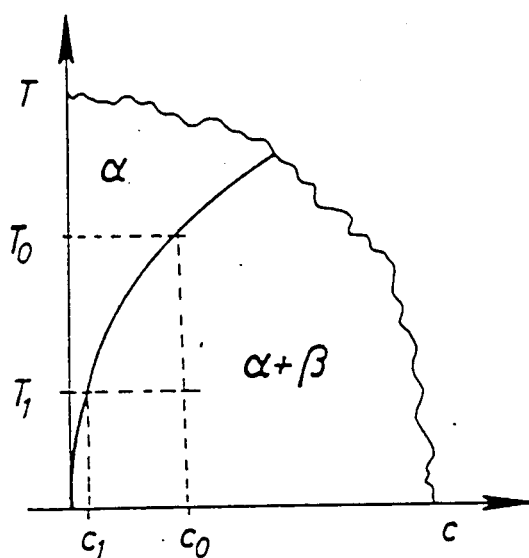


Fig. 12 Section of a binary equilibrium diagram with limited solid solubility [22].

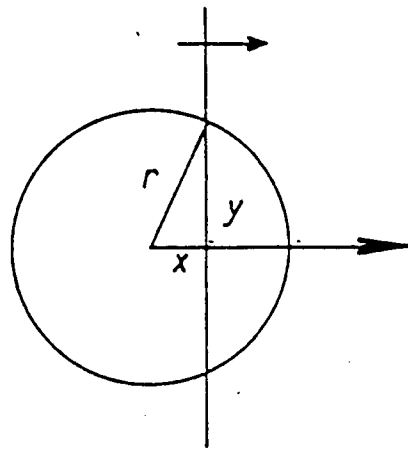


Fig. 13 Interaction between grain boundary and spherical inclusion [22].

than $\sim 0.1 \mu\text{m}$, the dislocations were formed as loops, often dispersed through the matrix. However, for larger particles, the dislocations were presented as local lattice rotations next to the particles. Therefore, during recrystallization, formation of low and high angle boundaries and boundary velocity could be determined by the kinetics of particle growth.

If the precipitation of particles (secondary particles) occurred before recrystallization, then the rearrangement of dislocations would be retarded, and therefore the formation of recrystallization nuclei would be hindered [30]; if the precipitation of particles occurred at grain boundaries, the velocity of grain boundary movement would be lowered, which is consistent with the results from Rodrigues et al. [17].

II.2. Mechanical Processing of 3xxx Al alloys

II. 2.1. Hot Rolling

Hot working is defined as deformation carried out above the recrystallization temperature and at strain rates in the range 0.5 to 500 s^{-1} [31]. Deformation at elevated temperature is different from that at room temperature, because recovery and even recrystallization (sometimes defined as dynamic recrystallization) occur simultaneously and substantially, so that little strain hardening takes place, especially in 3xxx Al alloys.

Mechanisms of plastic deformation of Al alloys during hot working have been extensively studied [32].

Hutchinson et al. [33] confirmed by transmission electron microscopy (TEM) that the matrix of most as-hot rolled specimens for 3004 Al alloy consisted of both recrystallized and unrecrystallized regions, the latter having well defined subgrain structures. Optical and scanning electron microscopy (SEM) observations also showed this phenomenon. They also found out that there was a maximum hot rolling temperature which corresponded to highest cube texture (Chapt. II.3.2.) after batch annealing with a heat-up rate of 94 °C/hour to 375 °C and holding for 1 hour, seen in Fig. 14 [33].

Therefore, tight control of temperature, strain rate, alloy content and particle size and distribution is essential at the high temperature deformation of Al sheet processing to optimize mechanical properties, and to lower the earing occurrence of the final cold rolled sheet.

II. 2.2. Cold Rolling

Plastic deformation which is carried out in a temperature range below the recrystallization temperature is often called cold working. Cold working increases the strength by strain hardening.

As mentioned before, Mn in 3xxx series alloys is largely distributed as dispersed Al-Mn (with other elements) intermetallic compounds, and Mg is mainly in solid solution in the Al matrix. Both Mg and Mn increases strain hardening greatly.

Substructure development in rolled materials affects both the mechanical strength and rolling texture characteristics. During cold rolling, tangles of dislocations can be interconnected progressively

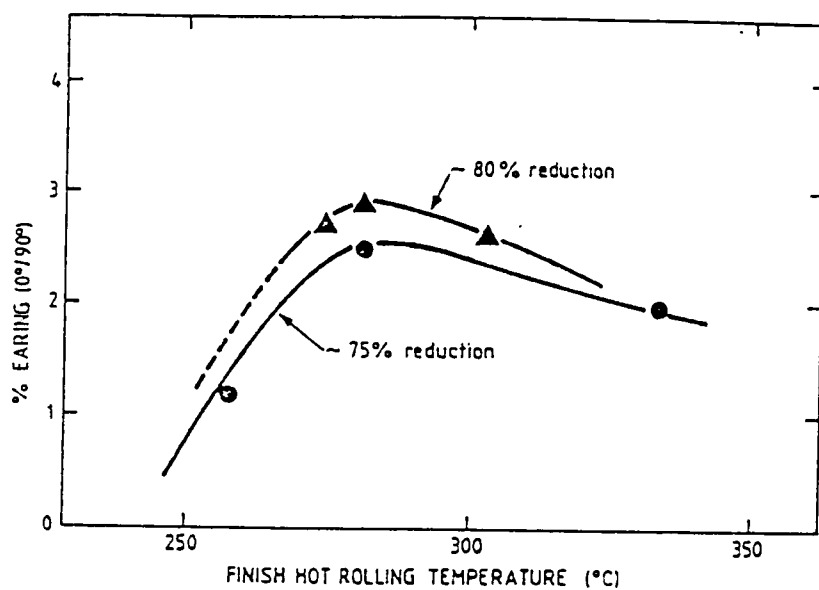


Fig. 14 Effect of finish hot rolling temperature on earing level of batch annealed (BA) hot bands, with homogenization S* and typical iron content for alloy 3004 [33].

* Homogenization S means higher temperature homogenization [33].

into a kind of rough cellular structure, so-called "cell structure" [34]. For 3004 aluminum alloy [34], when the reduction is about $\epsilon=0.1$ ($\epsilon=\ln(l_i/l_0)$, true strain; l_i is the instant length and l_0 is the original length during the standard tensile test) and above, a cell structure starts to form and grow, both in parallelogram-type and equiaxed-type; when the reduction is $\epsilon=0.2$ and above, microbands (MB) form and develop in the grains that have parallelogram-type cellular structure; when $\epsilon=0.3-0.9$, certain grains with equiaxed cellular structure are subdivided into deformation bands (DB) with large misorientations, whereas, some of the grains changed to cube oriented grains; finally, when $\epsilon=1.2$, the thick walled cell structure is eventually replaced by subgrains with 2-dimensional boundaries, and shear bands (SB) begin to appear and gradually play a more important role in the deformation process. These are illustrated in Fig. 15 [34], in which RP stands for rolling plane.

Furthermore, Liu et al. [34] pointed out that the deformed substructure in cold rolled 3004 due to inhomogeneous deformation should be described by microbands and deformation bands which are in turn characterized by the cell structures in the grains, if $\epsilon \leq 1.2$. They also concluded that the substructure in cold rolled 3004 was more likely that of the rolled structure in Cu than that in pure Al. In other words, cold rolled 3004 has a Cu type rolling texture rather than pure Al type rolling texture, which is similar to brass type texture. (Rolling texture will be discussed in Chapt. II.3.1.) This phenomenon may be explained by the fact that in cold rolled pure Al deformed under the same condition as 3004, microbands and

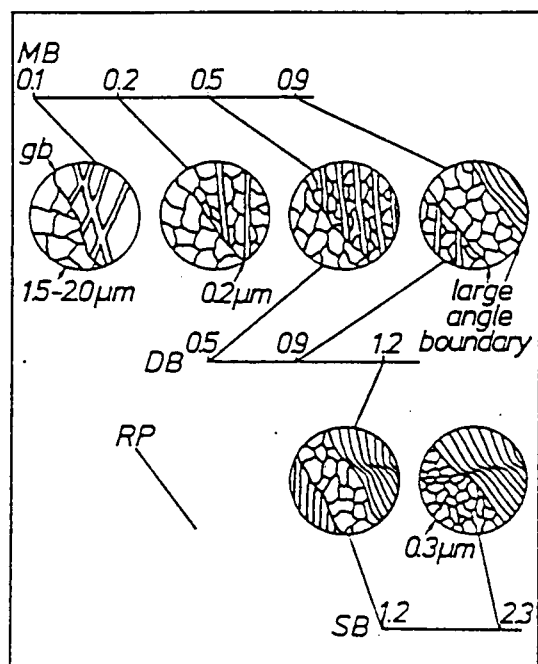


Fig. 15 Substructure evolution in rolled 3004 [34].

deformation bands were not found and the shear bands were less intense. This may also imply that the high stacking fault energy, therefore, the low separation of the partial dislocations, facilitated easy cross-slip and enhanced rates of dislocation climb compared to materials of relatively low stacking fault energy in which the partial dislocations are more widely extended.

II. 2.3. Deep Drawing

For 3xxx aluminum alloys, deep drawing is the final mechanical forming process in can-body manufacture in most cases.

The principal problem associated with the deep drawing of the can body is the formation of ears on the top of the cup, Fig. 16 [13]. The height and location of the ears on the drawn sheet vary with the texture of the sheet. Due to rolling texture (or "R" texture), four ears are located 45° to the rolling direction. The height of the ears increases roughly in proportion to the strength and intensity of the rolling texture.

The R-value, or the plastic strain ratio*, along with ΔR and $\bar{R}$ **, has great significance for the investigations of plastic anisotropy during deep drawing. For sheets of low-carbon steels, R-value has

 * $R = \epsilon_w / \epsilon_t$, ϵ_w is true strain along the width, ϵ_t is true strain along the thickness.

** ΔR , called "the planar anisotropy", $= (R_0 - 2R_{45} + R_{90})/2$; $\bar{R}$, called "the normal anisotropy", $= (R_0 + 2R_{45} + R_{90})/4$ [35].

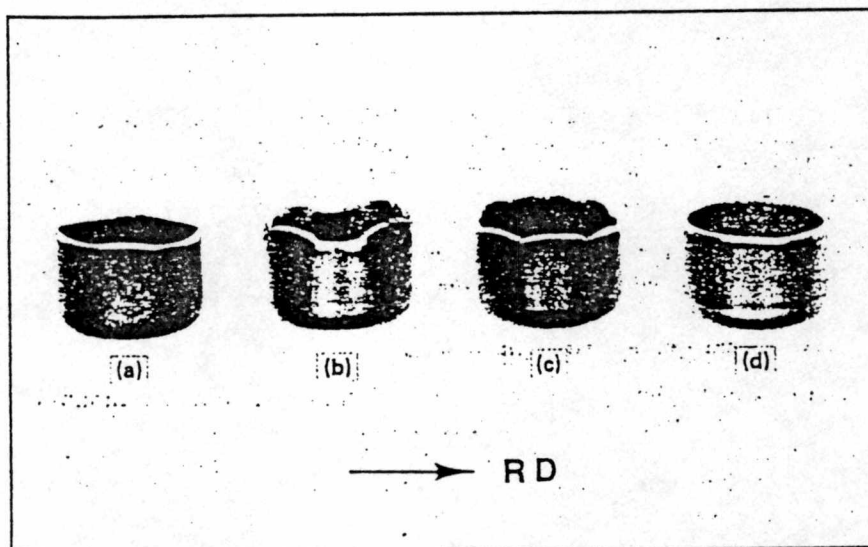


Fig. 16 Drawn aluminum cups (57% reduction) showing (a) 90° earring, (b) 45° earring, (c) mixed 90° and 45° earring, and (d) no earring, with respect to rolling direction, RD, shown in the arrow [13].

been used to correlate the anisotropy with texture problems, and shown great capability in improving the drawability in industry [36]. R-value affects earing percentage in Al and other certain materials [37].

II.3. Preferred Orientation and Texture in 3xxx Al Alloys

If an appreciable portion in each grain of an alloy (or metal) is aligned in certain preferred directions, the alloy is said to have developed a preferred orientation or texture. In crystalline materials, extensive plastic deformation creates preferred orientations. During deformation, while the grains are stretching, they also rotate so that certain crystallographic directions and planes gradually become aligned in a way that is characteristic of the type of deformation process used [38].

Preferred orientations can be determined by many ways [39-42], such as classical methods of pole figures, inverse pole figures, back-reflection and transmission Laue method, $K\alpha$ radiation methods and etching techniques [39]. Also ODF (orientation distribution function) method [40], stereoscopic presentation of Rodrigues vector [41], ultrasonic method and neutron diffraction measurements [42] are gaining their popularity nowadays. The most commonly used way is the pole figure, although it sometimes is tedious and costly to get.

Textures formed during rolling and after annealing are obviously different, and therefore, often termed as rolling textures and annealing textures or recrystallization textures.

II.3.1. Rolling texture

In 3xxx Al alloys, rolling textures are typically characterized by the copper type [43]. Such a texture may be described by a spread of orientations centered from $\{110\} \langle 112 \rangle$ (brass type) through $\{427\} \langle 232 \rangle$ to $\{112\} \langle 111 \rangle$ (pure copper type) as shown in Fig. 17 and Fig. 18* [43].

Hu [44] pointed out that the copper type can be described as having approximately $(123) [\bar{4} \bar{1} 2] + (146) [\bar{2} \bar{1} 1]$ as its principal ideal orientations. However, the pole figure of his was almost the same as that of Sheppard et al. [43]. Fricke et al. [45] summarized the texture components in aluminium alloys as Table 2 shows (they described the copper type texture component as $\{112\} \langle 111 \rangle$). In my opinion, $\{123\} \langle 412 \rangle + \{146\} \langle 211 \rangle = \{427\} \langle 232 \rangle$ in nature, because the distribution of orientations is almost continuous over a range, so picking an ideal orientation is largely arbitrary. Similar situation happened in Ito's paper [20], in which he gave a description of rolling texture of aluminum alloys as the orientations presented by a skeleton line running from the brass type $\{110\} \langle 112 \rangle$ through the S-type $\{236\} \langle 322 \rangle$ (yet, Fricke et al. [45] used $\{123\} \langle 634 \rangle$ as S texture, seen in Table 2, which means that many investigators have described these textures in different texture indices) into the pure Cu type $\{112\} \langle 111 \rangle$.

 * NOTE: In the original paper, the " a) as rolled b) annealed " must be mistakenly printed, should be crossed out.

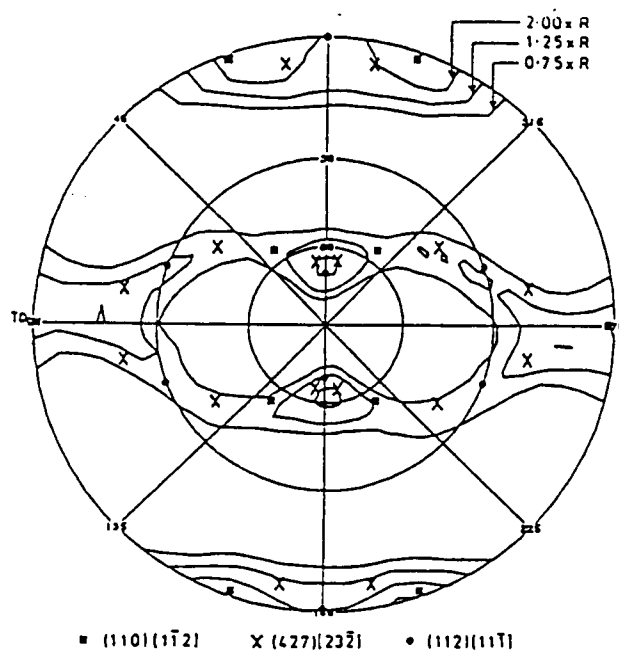
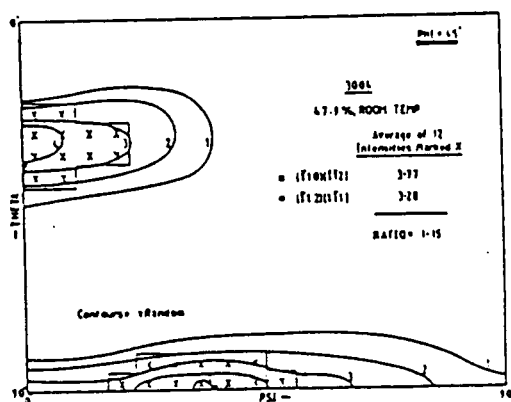
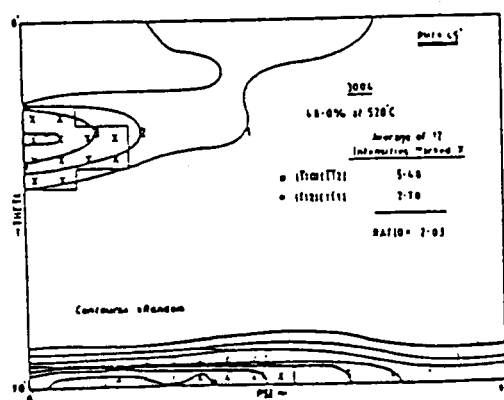


Fig. 17 (111) pole figure of AA3004 90% reduced at room temperature [43].



(a)



(b)

Fig. 18 Effect of temperature on the $\phi=45^\circ$ section of ODF in 3004 alloy [43]. (a) Rolling at room temperature, (b) rolling at 520 °C.

Table 2 Major components of texture [45].

Name	Indices
Brass	$\{110\}\langle 112\rangle$
S	$\{123\}\langle 634\rangle$
copper	$\{112\}\langle 111\rangle$
cube	$\{100\}\langle 001\rangle$
Goss	$\{110\}\langle 001\rangle$

Sun et al. [46], however, gave $\{124\}\langle 211 \rangle$ as the rolling texture in 3004.

It is favored in this thesis to use the definitions of Fricke et al. [45] as to describe the major rolling texture in 3xxx Al alloys.

II.3.1.1. The Mechanisms of Rolling Texture Formation

For the rolling textures of f.c.c. materials, there have been a lot of theories raised, which were summarized by Dillamore and Roberts [47]. Dillamore and Roberts [48] also found out by pole figures that commercial purity Al rolled at room temperature presented the brass type $\{110\}\langle 112 \rangle$ at lower reductions, the Cu type developed at higher reductions, and ideal pure Cu type $\{112\}\langle 111 \rangle$ only at very high reductions such as 98%.

Dillamore et al. [48, 49] theorized the cold roll texture in f.c.c. metals as $(110)[\bar{1}12] \rightarrow (153)[\bar{1}12] \rightarrow (362)[\bar{3}47] \rightarrow (121)[\bar{1}11]$, corresponding to three stages of stacking fault energy, γ/Gb^* . If γ/Gb is low, texture type would be $\{110\}\langle 112 \rangle$, if γ/Gb is high, texture type would be $\{112\}\langle 111 \rangle$, which was also pointed out by Leffers [50]. When if γ/Gb is intermediate, the texture type would be in the range of $\{135\}\langle 211 \rangle$ to $\{236\}\langle 734 \rangle$ [49].

 * γ , G , and b are respectively the specific surface energy of the stacking fault, the shear modulus of the material, and Burger's vector.

There have been a lot of arguments how the two different rolling textures have been evolved. They can be divided into two major groups. The first one favored that $\{111\} \langle 110 \rangle$ slip system was responsible, only by the different combinations of the $\{111\} \langle 110 \rangle$ slip, such as the cross-slip theory [48]. The other one thought that $\{111\} \langle 110 \rangle$ slip system could not account for even one of the two types of texture, therefore, some theories such as twinning, slip on planes rather than $\{111\}$ planes, and slip by partial dislocations were posed [48].

Leffers [50] favored the cross-slip theory in the first group, and by computer calculation of pole figures in $\{111\} \langle 110 \rangle$ cross slip subject to different stress systems, he plotted the calculated results into the experimental pole figure to compare them. The stress systems are shown in Fig. 19 [50]: *a*) the undeformed crystal; *b*) stress system I; *c*) stress system II; *d*) stress system III; *e*) stress system IV; *f*) stress system V; *g*) the combined stress systems II, III, IV, and V. RD, TD, and ND stand for rolling direction, transversal direction and normal direction respectively). Both brass type and copper type textures were presented. Fig. 20 shows the calculated pole figure for (III) + (IV) + random stresses for 80% reduction, and experimental pole figure for copper reduced 94% reduction superimposed on the same plot [*a*) 200 pole figure, *b*) 111 pole figure]. This agreement is quite impressive. Furthermore, he pointed out that if the cross-slip frequency was low, the dislocations could not escape from the pile-ups, therefore, the local stresses were

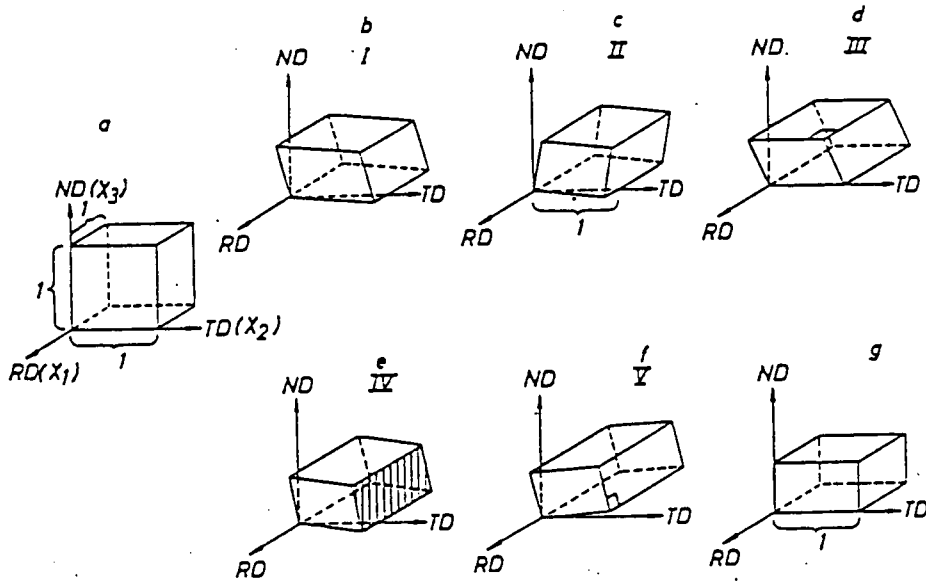


Fig. 19 The deformation of a cube-shaped crystal using the different stress systems [50].

- b) I: tensile stress σ in RD, compressive stress σ in ND, and $\sigma=\sigma$;
- c) II: an tensile stress δ added in the TD, to keep the edge in TD being 1;
- d) III: shear stresses τ_{12} and τ_{21} added, to preserve the right angles between the edges in the rolling plane (RP);
- e) IV: shear stresses τ_{23} and τ_{32} added, to keep the side faces (hatched area) vertical to the TD;
- f) V: shear stresses τ_{31} and τ_{13} added, to keep the edges NOT in the RP being vertical to the RD;
- g) : the combination of II, III, IV, and V states.

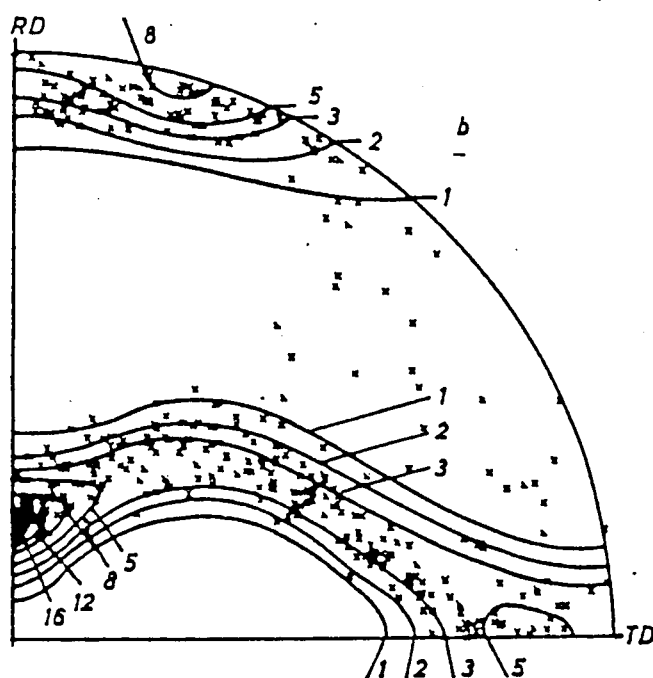
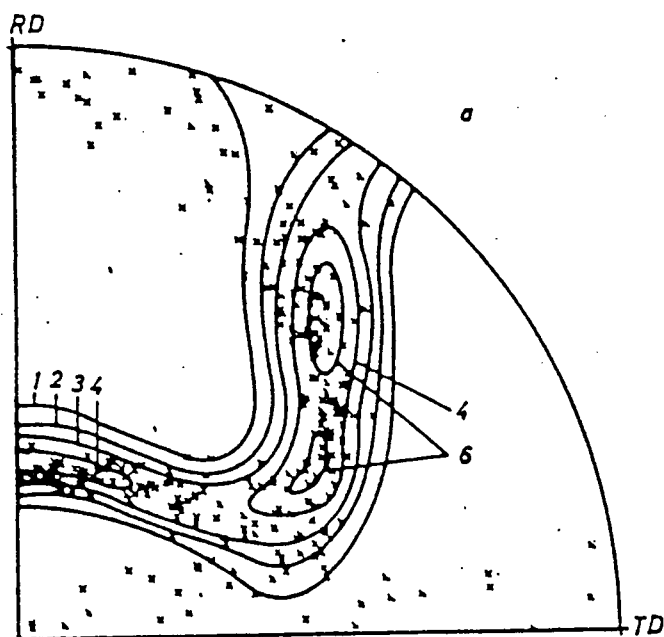


Fig. 20 Calculated pole figures for (III) + (IV) + random stresses, 80% reduction, and experimental copper type pole figures, 94% reduction, a) {200}, b) {111} pole figures [50].

relieved by special grain-boundary slip, and thus brass type texture was developed. If the cross-slip frequency was high, the dislocations could escape from the pile-ups, therefore, the local stresses were evenly distributed by cross slip, and thus pure copper type texture was developed.

Most recently, there are quite a few theories and models to evaluate and depict the rolling texture formation in terms of ODF, and the summary of them can be seen in Hollinshead and Sheppard's work [32]. It is proved that the tendency from brass type to copper type as the deformation increases is consistent with the results from former theories. However, the transition texture component variations are so different that none of the theories and models could individually explain them.

All the theories, however, need to be modified before satisfactory results could be reached in terms of texture calculations and experimental verifications.

II.3.1.2. Cold Rolling Texture of 3004 Al Alloy

For cold rolling, Hollinshead and Sheppard recently [32] summarized that at low reductions in 3004 Al alloy, texture components might have $\{110\}$ planes parallel to the rolling plane but with various directions parallel to the rolling direction, so called brass type, or " α fiber" [51]. As reduction increased to 90 to 98%, however, the texture component moved toward the copper type, or " β fiber" [51]. Here Hollinshead and Sheppard [32] said $\{326\}\langle 232 \rangle + \{225\}\langle 554 \rangle$ would be nearly equal to copper type or " β fiber".

II.3.1.3. Hot Rolling Texture of 3004 Al Alloy

For hot rolling, Sheppard et al. [43] showed from the pole figure point of view that 3004 subjected to multi-pass hot rolling at temperatures between 250 °C and 500 °C (reduction was about 50%) would have the copper type texture, and altering temperatures appeared to have little effect on the texture finally obtained, which meant that the strength of texture produced in the early passes of a rolling schedule was not influenced by the rolling conditions. By using ODF analysis, however, they did find out the effect of temperature, seen in Fig. 18.

They [43] finally concluded that the rolling temperature might have an effect on texture, but it was very difficult to detect and to understand the significance of the effect by observation of normal pole figures. In Fig. 21 were given the ODF results and the ratio of average of 12 intensities (seen in Fig. 18) about $(\bar{1}10)[1\bar{1}2]$ over $(\bar{1}12)[1\bar{1}1]$ on $\phi = 45^\circ$ section of ODF against reduction. Hollinshead and Sheppard [32] concluded that at the rolling temperatures of 400 °C and 520 °C, the effects of rolling quenching and interpass recrystallization affected the strength of the deformation texture and modified the balance of texture components present.

Hot rolling also increases the cube texture (see next chapter) component [32]. This is seen in Table 3. Here the cube strengths were detected by ODF method. It is seen that the cube texture strengths at rolling temperatures of 20 °C and 220°C are roughly below random intensity, yet, at rolling temperatures 400 °C and 520 °C are significantly above random levels. This could be due to the interpass

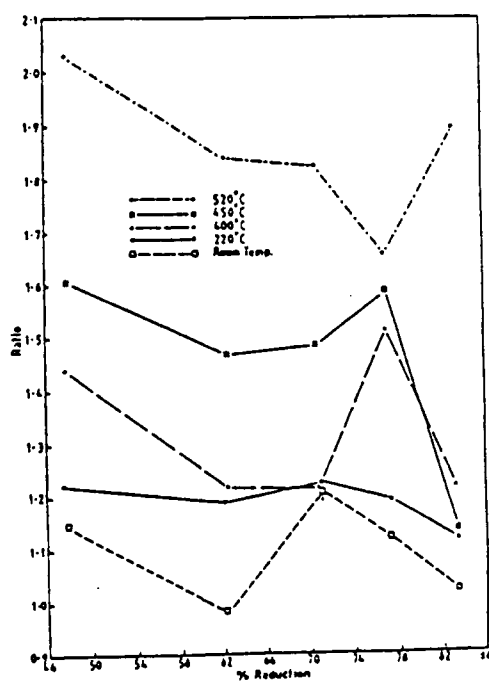


Fig. 21 Ratio of average of 12 intensities about $(\bar{1} \ 1 \ 0)[1 \ \bar{1} \ 2]$ over $(\bar{1} \ 1 \ 2)[1 \ \bar{1} \ 1]$ on $\phi = 45^\circ$ section of ODF against reduction [43].

Table 3 Cube texture strengths in as-rolled material [32].

rolling temperature	reduction(%)							
	48	63	71	77	83	90	96	98
20 °C	1.0	1.1	0.5	0.6	0.4	0.3	0.6	0.8
220 °C	0.8	0.9	0.4	0.4	0.5	0.7	--	--
400 °C	0.9	1.2	0.4	1.4	1.4	1.7	--	--
520 °C	1.8	1.6	2.6	1.6	2.9	--	--	--

recrystallization effects at 400°C and 520 °C.

II.3.2. Annealing Texture

Annealing texture, sometimes also called recrystallization texture, is defined as the texture formed by recrystallization when new grains are formed during annealing the cold worked material, characterized by large angle grain boundary motion. Because the recrystallization texture is different from the deformation texture, this reorientation has been extensively studied in rolled sheet and varies considerably with the fabrication history and the composition of the alloy [52]. There is a strong tendency for the new grains to form with a cube plane $\{100\}$ parallel to the surface of the sheet and a cube edge parallel to the rolling direction (often called cube texture). In contrast to rolling texture, which produces 45° earing during deep drawing, this $(100)[001]$ (cube) texture produces four ears at the 0° and 90° positions around the drawn cup.

Besides cube texture, there exist other components in the annealed material, for example, Goss texture component $\{110\}\langle 001\rangle$, and R component (some literature calls it retained rolling texture) $\sim\{123\}\langle 412\rangle$ [32], which are considered to be one of the contributions to the final annealing texture. Table 4 [33], shows the peak intensity of cube, Goss, and R components, with a large fraction of random spread in each treatment.

Table 4 Measurements of texture (ODFs) for various alloy treatments [33].

Alloy*	Hom.**	Anneal.***	<u>Peak intensity, x random</u>			Vol. fraction in random, %
			Cube	Goss	R	
LFe	M	BA	27.9	10.2	4.6	56
	M	CA	16.2	9.0	3.1	66
NFe	M	BA	21.6	7.9	3.7	55
	M	CA	15.4	8.3	2.9	68
	S	BA	11.8	4.7	2.4	64
	S	CA	1.2	5.1	2.5	67
HFe	M	BA	16.0	6.8	3.7	69
	M	CA	15.2	5.9	3.2	68
	S	BA	11.1	4.4	2.5	64
	S	CA	10.5	4.2	2.6	69

* LFe, NFe, and HFe represent, respectively, low, normal and high Fe content in the 3004 alloy, according to the author [33].

** M, and S represent, respectively, modified and standard homogenization temperatures [33].

*** BA and CA represent, respectively, batch anneal and continuous anneal, the former, a slower heat-up rate than the latter [33].

Nes [24] pointed out that the following factors would influence the cube texture formation: the particle content; elements in solid solution; rolling temperatures; etc. The effect of elements in Al can be seen in Fig. 22. The diagram illustrates the general trend in the variation of the strength of the cube texture component with alloy content. The dotted area reflects scatter in the results.

The influential factors on cube texture formation for Al alloys are sketched in Fig. 23 [24]. Here we can see that the strength of cube texture and the grain size depend in the same way on the same variables. For example, as the number of primary particles increases, both grain size and strength of cube texture decrease, and as the Zener drag pressure of secondary dispersoids increases, both grain size and strength of cube texture increase, and so on.

Sundberg [53] concluded from his and other's work that if the (110)[112] rolling texture was dominant, the main recrystallization texture would be Goss {110}<001> type. When the normal rolling texture was a {112}<111> type, the annealing texture would be cube texture dominant, with other orientations in it.

Oriented nucleation and preferred (or oriented) growth of certain grains, the two major mechanisms which have been studied in the last few decades, are thought to be responsible for the cube texture formation during recrystallization in f.c.c. metals. Most recently Hu [54] quite thoroughly reviewed all this.

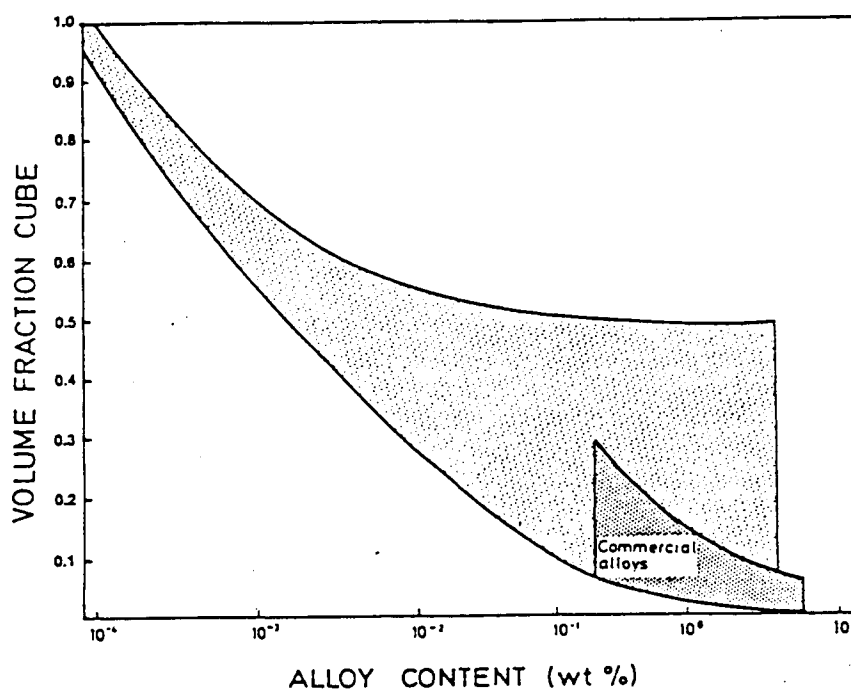


Fig. 22 This diagram illustrates the general trend in the variation of the strength of the cube texture component with alloy content for 3xxx alloys [24] .

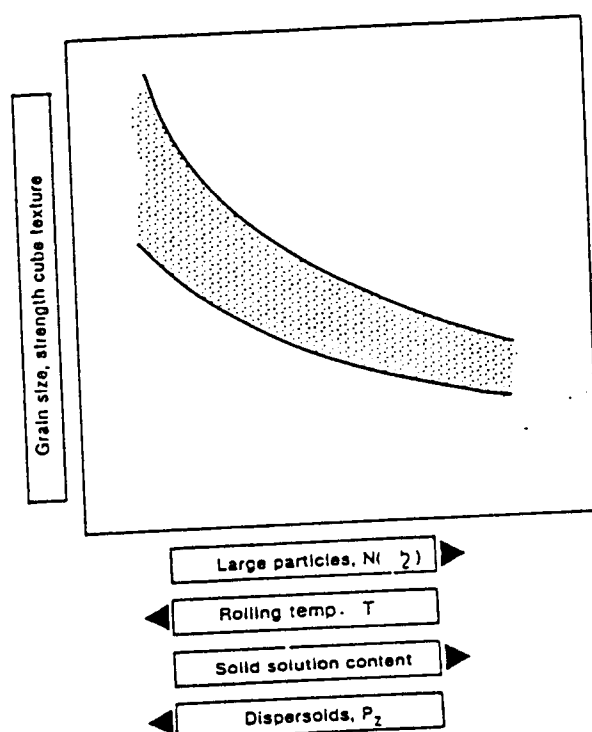


Fig. 23 The recrystallized grain size and the strength of the cube texture component as a function of the parameters given [24].

EXPERIMENTAL PROCEDURE

III.1. Material for the Experiment

The material, 3104 aluminum alloy, was provided by Ravenswood Aluminum Corporation. The chemical composition is in Table 5. Properties of 3104 alloy can be seen in the following [6]. At H19 state, 3104 has the typical following properties, compared with 3004:

	3104 alloy	3004 alloy
Tensile strength:	290 MPa (42 ksi);	295 MPa (43 ksi)
Yield strength:	260 MPa (38 ksi);	285 MPa (41 ksi)
Elongation:	4%	2%

The as-received material was hot-rolled in the plant to a thickness of 5.03 mm (0.198 in) and air cooled. The sheets of the material were randomly coded in alphabetic way for the testing convenience. The sample plate size for rolling and annealing was about 350 mm x 250 mm (14 in x 10 in).

The heat treatments which were given to the 3104 alloy are based on processes of interest in producing can stock, seen in Table 1. The properties measured and the microstructural features to be characterized were designed to indicate the factors important in controlling earing.

III.2. Cold Rolling and Annealing

Five practices (Table 1) were tested in the research. Cold rolling

Table 5 Composition of 3104 Al alloy (compared with the nominal composition of 3104 and 3004).

Alloy	element(wt%)				
	Cu	Mn	Mg	Fe	Si
3104	0.20	1.0	1.15	0.43	0.23
3104*	0.15	1.1	1.0	~	~
(nominal)					
3004*	~	1.2	1.0	~	~
(nominal)					

* From Metals Handbook [6].

was carried out on our departmental 203 mm (8 in) diameter rolling mill. For each individual pass, a reduction of 0.03-0.1 mm (0.001-0.003 in) was used, depending on the thickness and the surface condition, to reach the required reduction. Two reductions, 35%, and 88% were used in the intermediate and final rolling conditions.

The temperature distribution throughout the sample plates was found by McGill-Taylor [4], and it showed a uniform distribution, only $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$) in variation because of the high thermal conductivity of aluminum.

For the slow heat-up annealing (sometimes called batch annealing), which was $14^\circ\text{C/hr} \pm 0.5^\circ\text{C/hr}$ ($25^\circ\text{F/hr} \pm 1^\circ\text{F/hr}$), the furnace control was investigated by McGill-Taylor [4]. For fast heat-up annealing (sometimes called strip annealing), the rate was $140^\circ\text{C/min} \pm 5^\circ\text{C/min}$ ($252^\circ\text{F/min} \pm 9^\circ\text{F/min}$) at first 3 minutes, and $40^\circ\text{C/min} \pm 2^\circ\text{C/min}$ ($72^\circ\text{F/min} \pm 4^\circ\text{F/min}$) in the following 4 minutes. For both batch and strip annealing, the sample plates were soaked at $332^\circ\text{C} \pm 2^\circ\text{C}$ ($630^\circ\text{F} \pm 4^\circ\text{F}$) for two hours. Figs. 24 and 25 give the schematic plots of the slow heat-up (batch annealing) and fast heat-up (strip annealing) processes.

This temperature control was done on a programmable temperature controller (Omega Series CN-2010). The furnace temperature was plotted using an Omega Model # RD-2062 chart recorder with a thermocouple module. A typical heating curve is shown in Fig. 26.

In order to achieve the fast heat-up rate, two fairly thick metal plates (about 50 mm) were hinge-connected with a handle on top, and kept in the furnace for sufficient time to reach the required

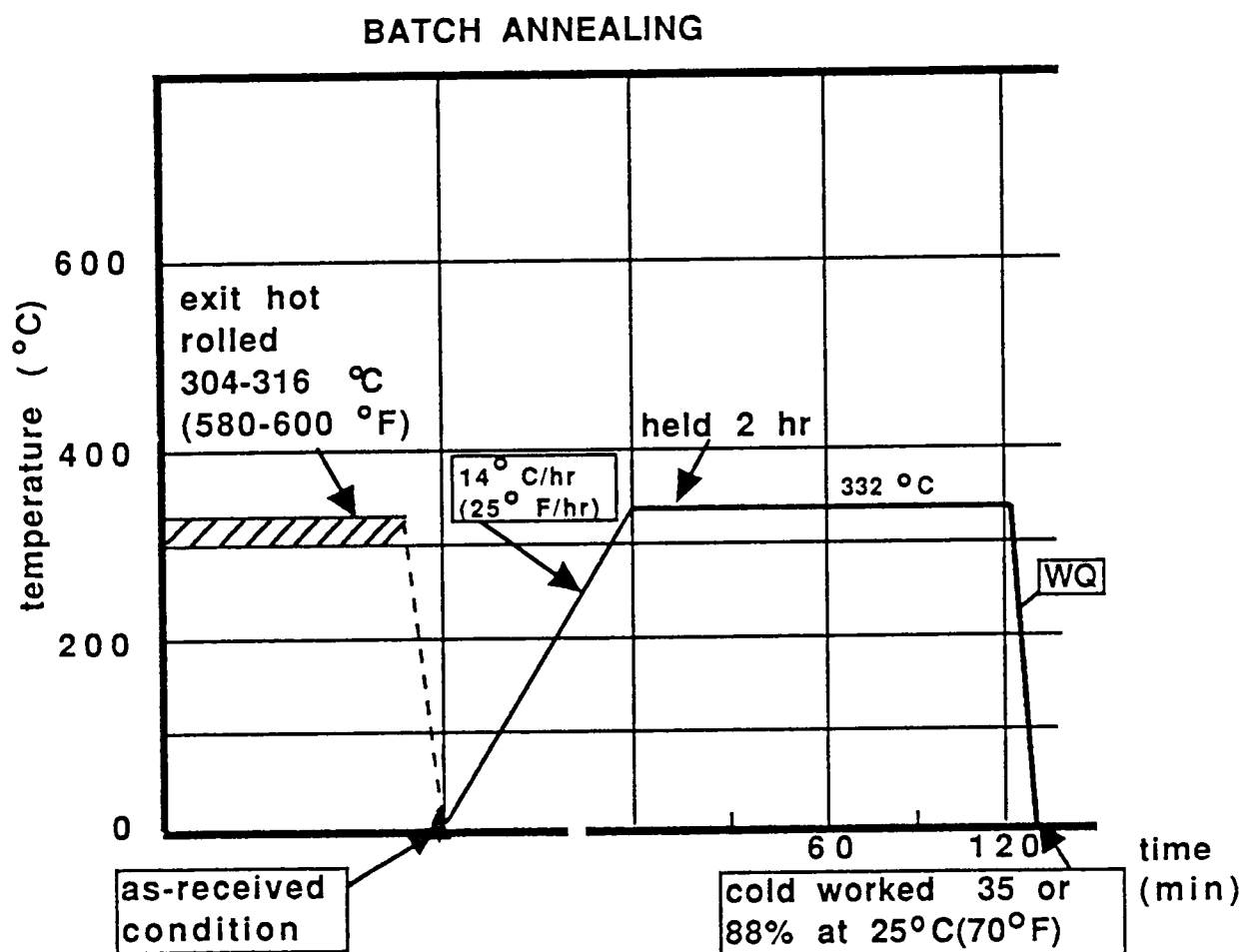


Fig. 24 Schematic plot of slow heat-up rate annealing, so-called batch anneal (BA). WQ stands for "water quenched".

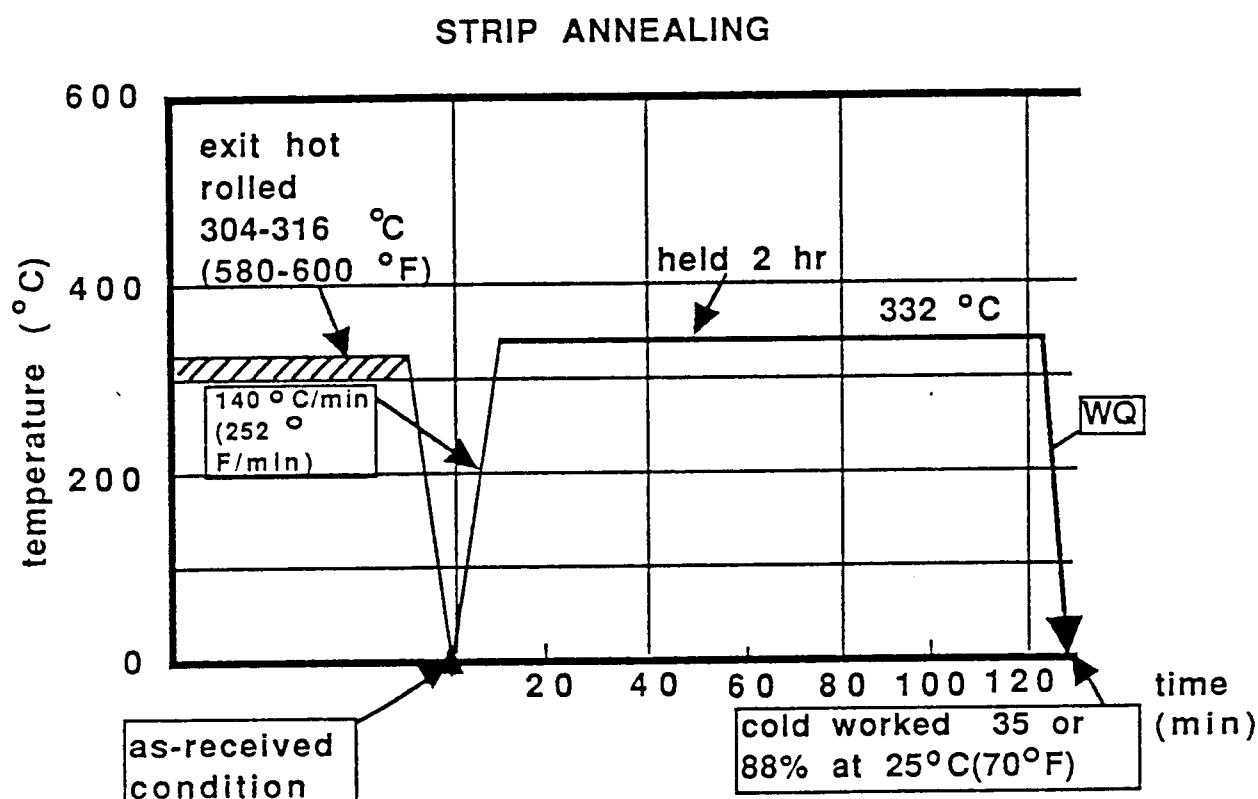


Fig. 25 Schematic plot of fast heat-up rate annealing, so-called strip anneal (SA). WQ stands for "water quenched".

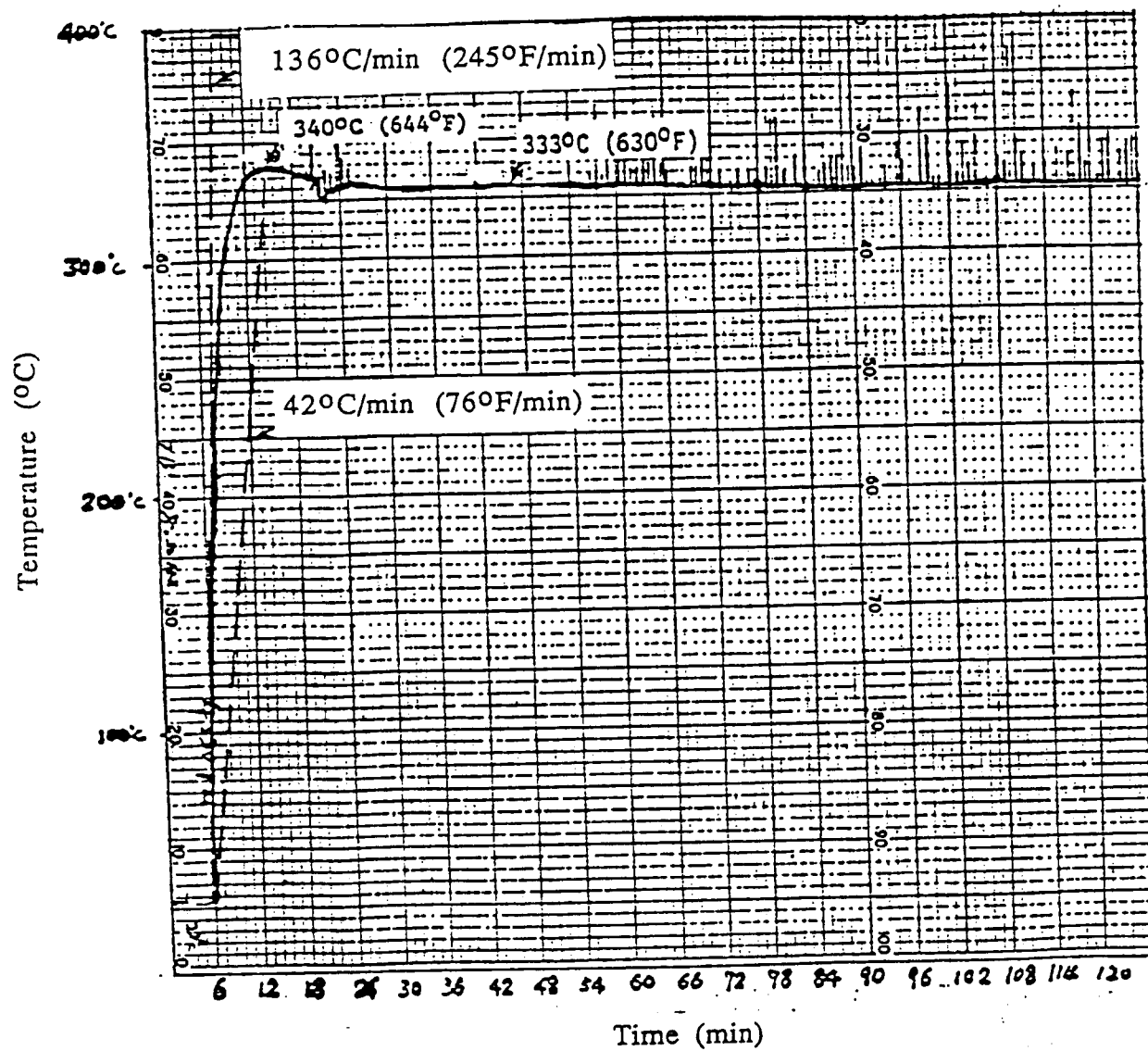


Fig. 26 A recording of the fast heat-up rate annealing.

temperature. The test plate had the same thickness as the real sample plate and had welded to a number of K-type 24 gauge duplex insulated thermocouples (by tungsten inert gas-welding technique). Then it was put in between the preheated thick plates. By adjusting the setup temperature and the furnace door, finally the satisfactory heatup rate was achieved.

III.3 Mechanical Testing

III.3.1. Tensile Testing

Tensile bars after each step in every practice were cut at 0°, 45°, and 90° to the rolling direction. The size of the tensile bar was 1.9 cm x 25 cm (0.75 in x 10 in). All the samples were tested at Ravenswood Aluminum Corporation. The original test data are tabulated in Appendix 1.

III.3.2. Earing Cup Test

Samples after 35 and 88% cold rolling were also tested in earing percentage experiments, which was done at Ravenswood Aluminum Corporation. Test pieces (178 mm x 178 mm or 7 in. x 7 in.) were cut off the rolled plates, and the percent earing was examined and calculated by the formulæ [4]:

$$\text{he(average ear height)} = \frac{\text{he}_1 + \text{he}_2 + \text{he}_3 + \text{he}_4}{4} \quad \text{eq. 8(a)}$$

$$ht(\text{average trough height}) = \frac{ht_1 + ht_2 + ht_3 + ht_4}{4} \quad \text{eq. 8(b)}$$

$$\text{Earing}(\%) = \frac{he - ht}{[he + ht]/2} \times 100 \quad \text{eq. 8(c)}$$

Here he_i is the individual ear height and ht_i the individual trough height.

According to Ravenswood Aluminum Corporation, a stress relief is given to the cold rolled sheet before the earing cup test is conducted. The stress relief is at $^{\circ}\text{C}$ ($^{\circ}\text{F}$) for minutes. The original test data of earing is tabulated in Appendix 2.

Earing test results give both the earing positions on the cup tested in terms of the directions to the rolling direction and the earing strengths in each sample. Later on we will see if any correlation exists between earing test and other ways to reflect the texture.

III.4. Optical and SEM Metallography

Metallographic samples were cut off from the tensile bars, as shown in Fig. 27 [4]. MgO (magnesium oxide) powder must be used to fine-polish the samples [4]. Etching was conducted in an electro-etching method using Barker's reagent [55], 1.8% solution of fluoboric acid (HBF_4) in distilled water, as the electrolyte. 20 to 30 volts were applied for 60 - 90 seconds at room temperature.

In order to distinguish the two types of main primary particles

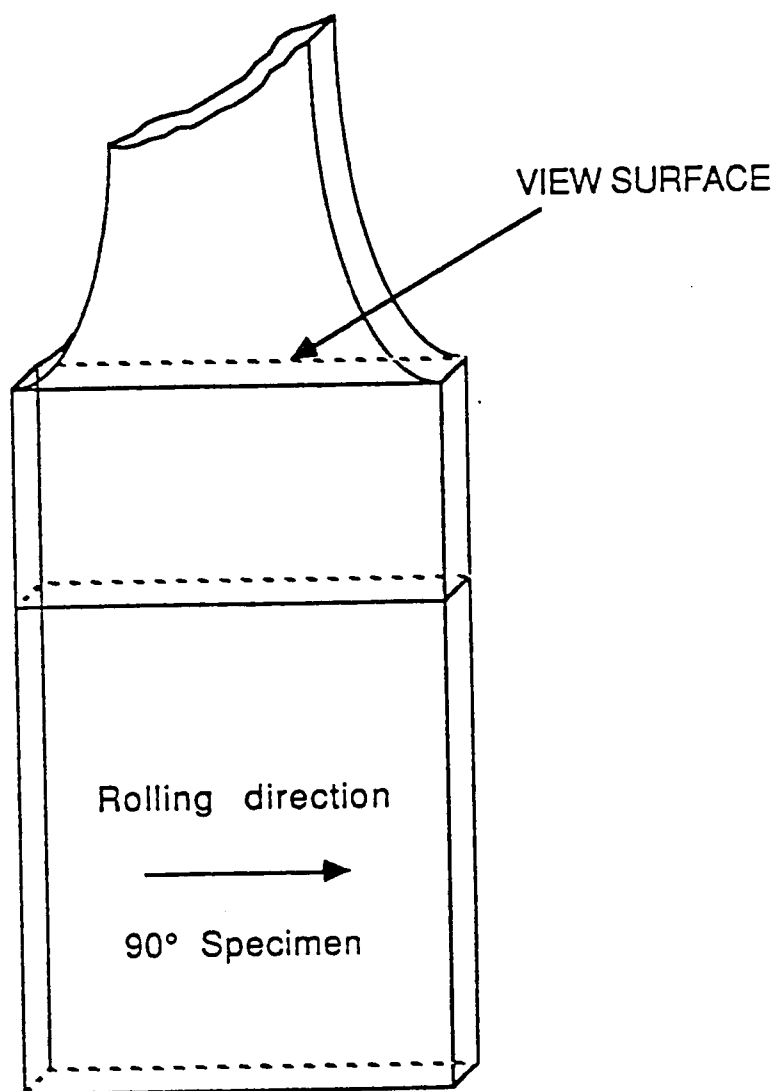


Fig. 27 Metallurgical samples cut off from the tensile bars [4].

in the microstructure, a special agent (90 ml of acetic acid, 5 ml of 1% HF and 5 ml of H_2SO_4) [33] was used. The α - $\text{Al}_{15}(\text{Fe}, \text{Mn})_3 \text{Si}_2$ particles etch dark and the $\text{Al}_6(\text{Fe}, \text{Mn})$ particles light.

All of the SEM examinations were carried out on a Cambridge S360 Scanning Electron Microscope. The detection of the primary particles and secondary particles was done in the SEM using a Link AN-10000 energy dispersive x-ray analyzer. Particle percentage calculations were conducted based on the SEM micrographs in the image processing method (IPM). And the particle distribution density was calculated based on optical and SEM micrographs.

III.5. X-Ray Diffraction Intensity Measurements

One of the ways to determine the textures in the rolled conditions used here was x-ray diffraction of certain planes in the samples [56]. The machine was a Rigaku Geiger-Flex diffractometer with $\text{Cu K}\alpha$ radiation target and Ni filter. A set-up voltage of 35 kV and current of 20 mA were used for each sample. The programmings for the different diffraction planes on the Rigaku diffractometer in this material are listed in appendix 3.

All the samples were cut off from the tensile bars and the earing cups in the "unchanged" portion, so that no further deformation could influence on the texture existed already (Figs. 28 and 29). For the cupping experiment, it was found by Ravenswood Aluminum Corporation that during cupping the grid pattern at the bottom of the cup did not change. Therefore we assume that the bottom of the ear cup is unaffected by the cupping. For most of the

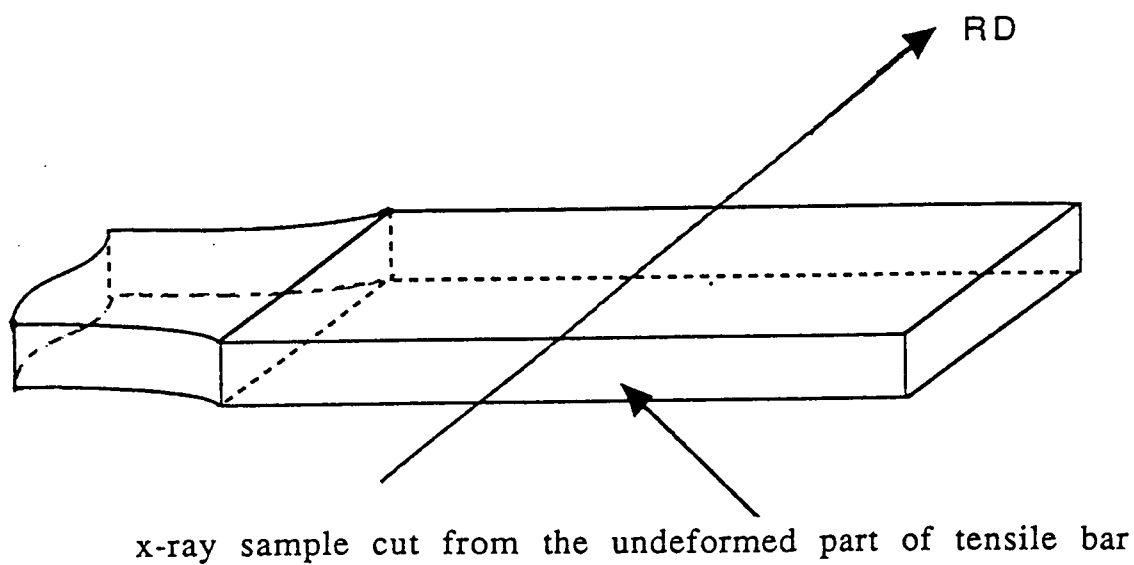


Fig. 28 X-ray samples cut from the tensile bars.

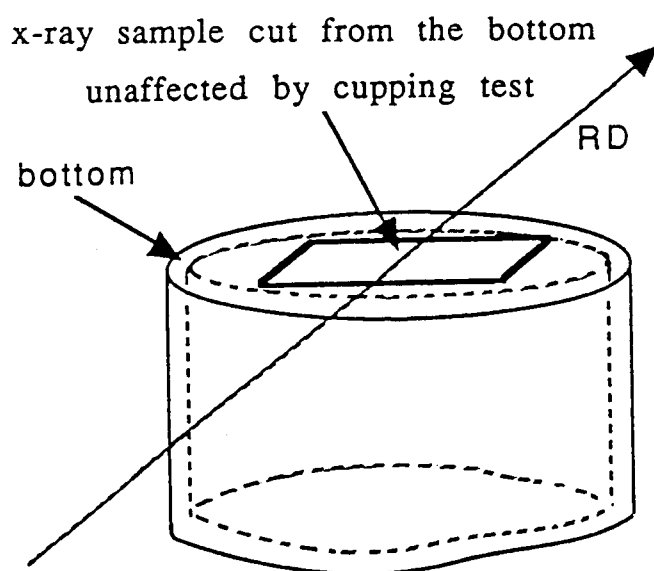


Fig. 29. X-ray samples cut from the earing cup bottom.

samples, the {111}, {200}, {220}, and {422} diffraction planes were examined.

One of the samples was also examined by multi-layer etch off method. This sample was examined by x-ray diffraction first, then a certain layer was etched off in a NaOH solution, and then examined again, etched again, etc. This treatment was repeated till the half thickness was reached to see any variation in the diffracted plane intensity.

The principle of x-ray diffraction is to get the intensities from certain diffracted plane in the rolled samples and in the standard random Al sample, then compare them, by using the following formula shown (for a (hkl) diffracted plane),

$$R = I_r / I_s \quad \text{eq. 9}$$

where

I_r - The X- ray intensity of rolled sample,

I_s - The X-ray intensity of standard random sample.

The standard Al sample was sintered by powder metallurgical technique in Oak Ridge National Laboratory.

III.6. Ultrasonic Testing

One of the most recent developments of ultrasonic technology in industry is to investigate texture problems in metal rolling procedures [41]. The principles of this technique are to measure the velocities of bulk waves, and of guided and surface waves in polycrystalline aggregates of cubic metals, then to correlate the variations of the velocities with polycrystalline texture in rolled

aluminium and steel sheet, assuming that other factors such as dislocations, second-phase particles, etc. having no direct influence on the velocities. The correlation is done by the use of orientation distribution function (ODF) coefficients, which have been one of the quantitative and reliable measures of the texture in the last twenty years. Good agreement has been reported between texture measurement and ultrasonic velocity result [41]. People are now looking forward to the application of the ultrasonic technique to the monitoring of texture during the on-line rolling production of the sheet because of the reliability and convenience in the near future.

Due to some of the limitations, however, only the 88% rolled samples were examined in this technique at the Engineering Mechanics Department, University of Kentucky, Lexington, KY40506.

III.7. Pole Figure

By far the most commonly used way to study texture issues in the materials science field has been the pole figure technique. The samples for pole figure in this research were the same ones for x-ray diffraction, only with the sample surface being chemically etched off about 0.2 mm (0.008 in). The pole figure experiments were carried out in the texture laboratory, Department of Materials Science and Engineering, University of Pittsburgh, Pennsylvania.

The Schulz reflection method (Fig. 30) [59] was employed for the pole figures, which was carried on a heavily modified GE XRD-5 x-ray unit and diffractometer. The modifications included the use of a Siemens texture goniometer and a Canberra pulse height analyzer,

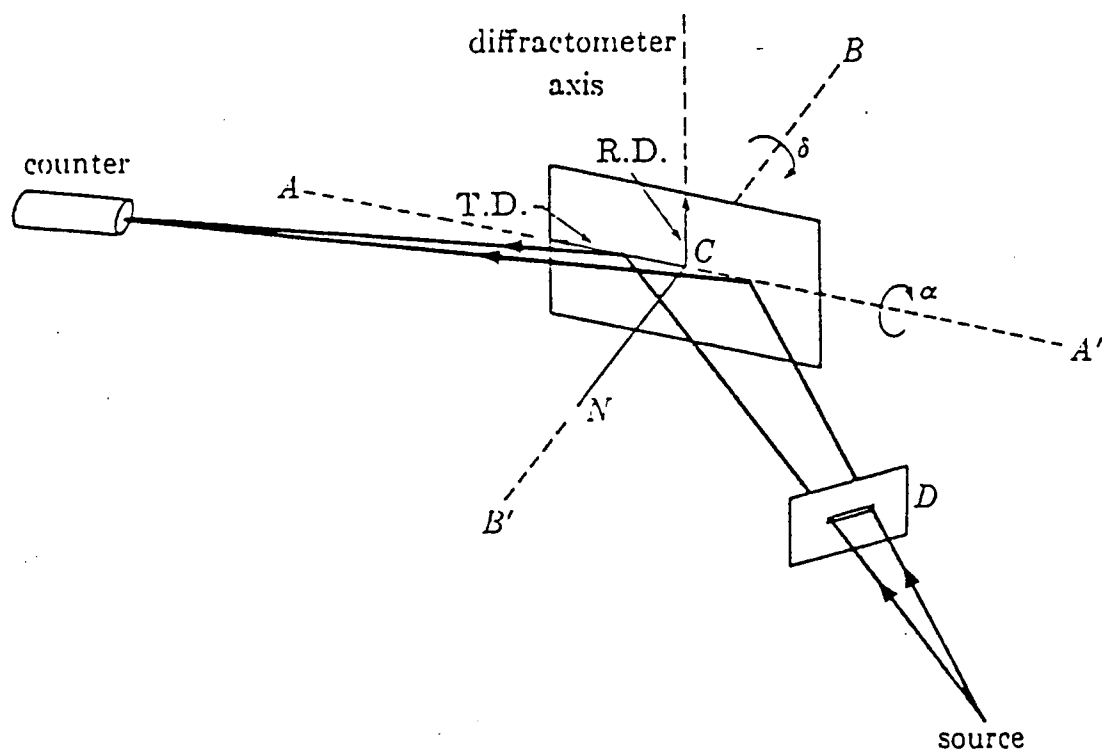


Fig. 30 Shulz reflection method for pole figure formation [59].

digital count rate meter and teletype interface. This enabled the pole figure to be computer plotted to fit a 300 mm diameter stereographic projection. Co $K\alpha$ radiation was used and coupled with a scintillation counter which was for high counting efficiency. The data were normalized against a random powder Al sample out to 80° tilt.

CHAPTER IV

RESULTS AND DISCUSSION

The effect of cold roll reduction and annealing on the tensile properties was well investigated by McGill-Taylor [4] for this material. Here it is the purpose to compare the tensile properties of every individual practice with each other and correlate the tensile properties with earing characteristics.

IV. 1. Mechanical Properties

Results from the tensile tests included the yield strength (YS), tensile strength (TS), and elongation (El. %). For each individual stage of a treatment, samples oriented 0° , 45° , and 90° relative to the rolling direction were tested and the data are in appendix 1.

The YS, TS and El. % data showed little difference in 0° , 45° , and 90° relative to the rolling direction for each individual stage of the treatment ($\leq \pm 10$ Mpa (± 1.5 ksi) for YS and TS, and $\leq \pm 1\%$ for El.), which is consistent with McGill-Taylor's work [4]. Therefore, the average value of the tensile properties in 0° , 45° , and 90° relative to the rolling direction for each individual stage of the treatment was used to plot Fig. 31. In Fig. 31, (a) reflects the "hot rolled" condition, or as-received condition (AR); (b), practice 1 (Prac.1); (c), Prac.2; (d), Prac. 3; (e), Prac. 4; (f), Prac. 5. Note that comparing Prac. 1 and Prac. 2 with the AR condition, both batch and strip anneal decreased

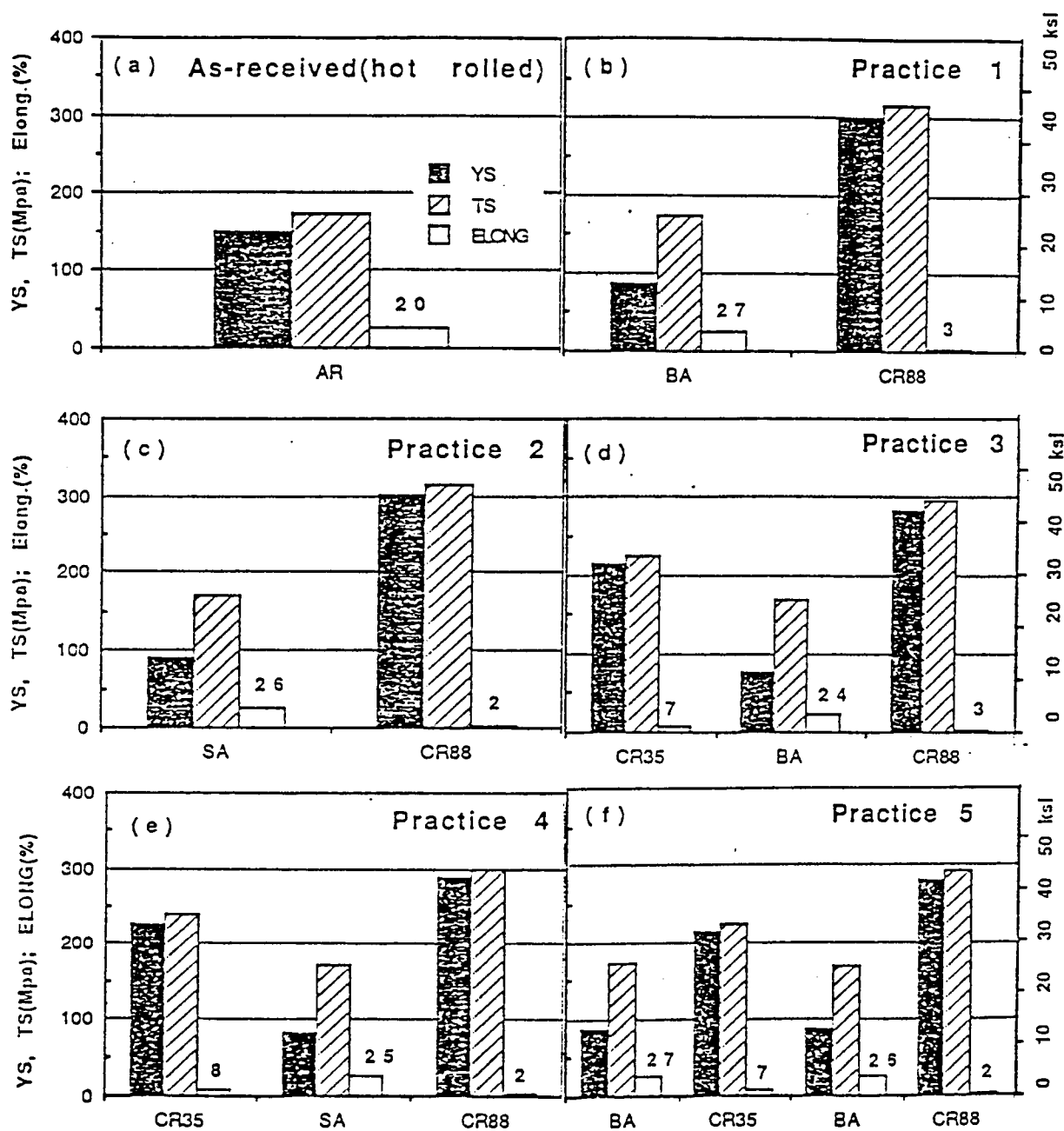


Fig. 31 The tensile mechanical properties at each stage in each processing practice.

the YS of AR condition substantially, increased the El. %, yet kept the TS the same. This means that the AR condition is not a fully hot rolled condition, since further recrystallization takes place afterwards upon both batch strip annealing. Microstructure examinations confirmed this (see IV. 2). It is also shown that the properties for Pracs. 1 and 2 are almost identical at each step, and that the properties for Pracs. 3 and 4 are essentially identical at each step, which means that heat-up rate did not affect the tensile properties. Comparing Prac. 3 with Prac. 5, intermediate BA gave the same properties, suggesting that the intermediate BA before CR35 did not give a significant effect on the final tensile properties.

Fig. 32 presents the effect of processing practice on the tensile mechanical properties after final cold rolling of 88%, where the data from 3 directions to the rolling direction (RD) are plotted in the same plot; however, there is no difference in properties with orientation in the plate, considering the experimental uncertainty of about ± 10 MPa (± 1.5 ksi) for YS and TS, and $\pm 1\%$ for El..

Results from the cup earing test can be seen in Appendix 2. And the results after the final 88% cold roll condition of the all 5 practices are shown in Fig. 33. The ears were at 45° to the RD in all of them, characteristic of that of highly cold worked 3xxx Al alloys.

Pracs. 1 and 2 gave lower earing, and Pracs. 3, 4 and 5 higher earing. Note that Pracs. 1 and 2 gave higher tensile strengths, and Pracs. 3, 4 and 5 corresponded lower tensile strengths, with all the practices having same tensile ductility.

It is obvious that the intermediate CR35 and annealing caused

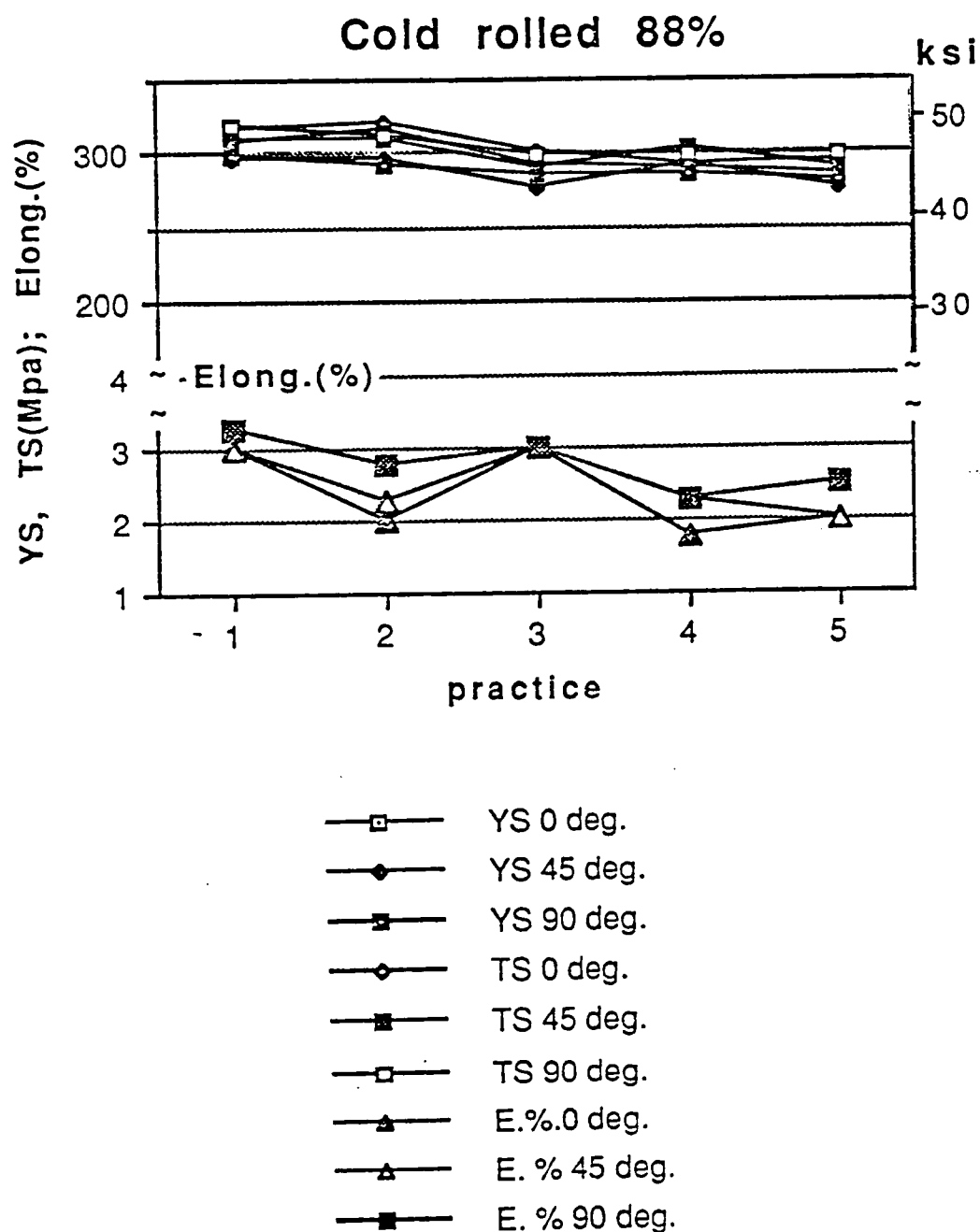


Fig. 32 The effect of processing practice on the tensile mechanical properties for the final 88% cold worked condition.

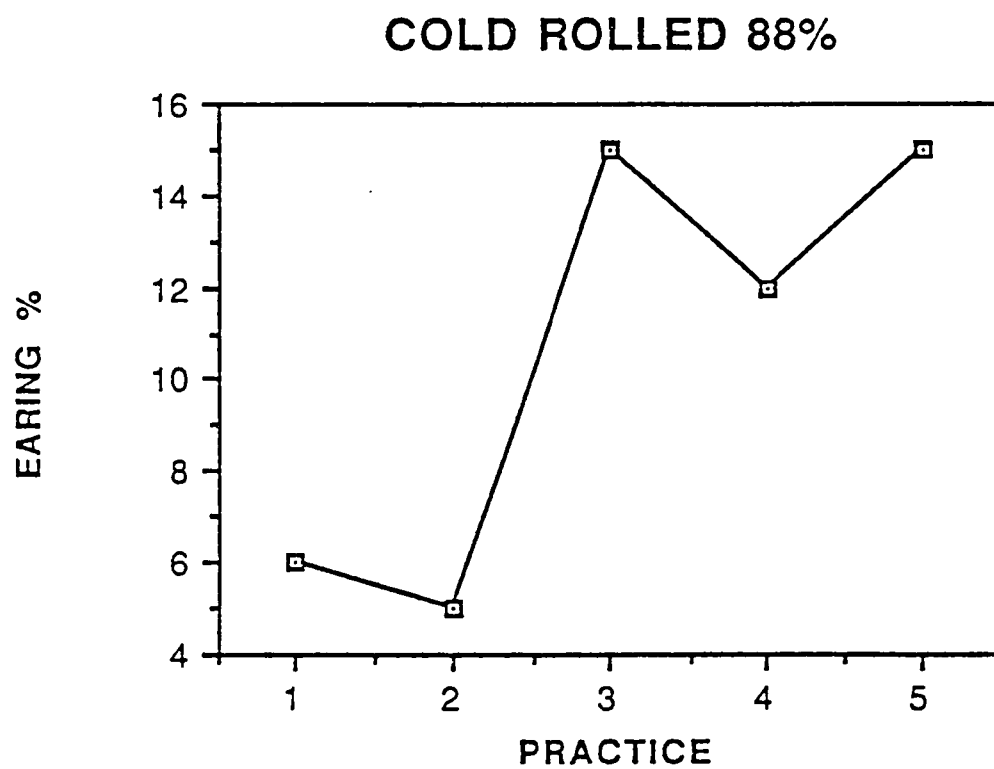


Fig. 33 The effect of processing practice on the degree of earring (45°) for the final 88% cold worked condition.

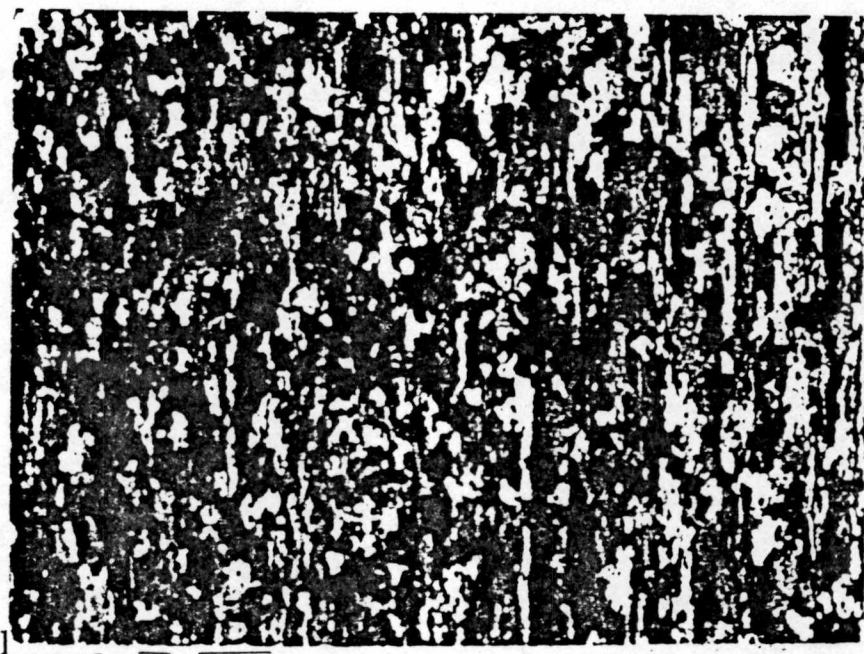
the higher earing and the CR35 condition is more dominant, because Pracs. 3 and 5 have the same earing strength. Although a treatment of CR20 + 332 °C (630 °F) 2 hrs. anneal could accomplish recrystallization [4], which is similar to my CR35 + 332 °C (630 °F) 2 hrs. anneal condition, this fully recrystallized structure gave much higher earing, comparing with that of fully recrystallized structure from Pracs. 1 and 2.

IV. 2. Microstructure and Particle Examinations

Fig. 34 shows the grain structure of the AR condition of Al 3104. Note that the edge of the plate shows equiaxed grains, Fig. 34 (a), whereas the center contains a mixture of equiaxed and elongated grains, Fig. 34 (b), which also verifies that the recrystallization was not complete (consistent with the results from tensile tests); in other words, both recrystallized and unrecrystallized regions exist in the hot roll condition [33].

Fig. 35 shows the grain structure of Prac. 1, and Fig. 36, Prac. 2. The final cold rolled 88% structures in both cases optically did not show any differences, even at higher magnification. However, the lower heating of the BA condition allowed the development of larger grains, comparing Fig 35 (a) with Fig. 36 (a).

Fig. 37 shows the grain structure of Prac. 3, and Fig. 38, Prac. 4. The similar effect was also observed that the slower heating of the BA condition favored the development of larger grains even after intermediate 35% cold roll, comparing Fig 37 (b) with Fig. 38 (b). Notice that post-annealed grain sizes in both BA and SA conditions



Edge of plate

(a)

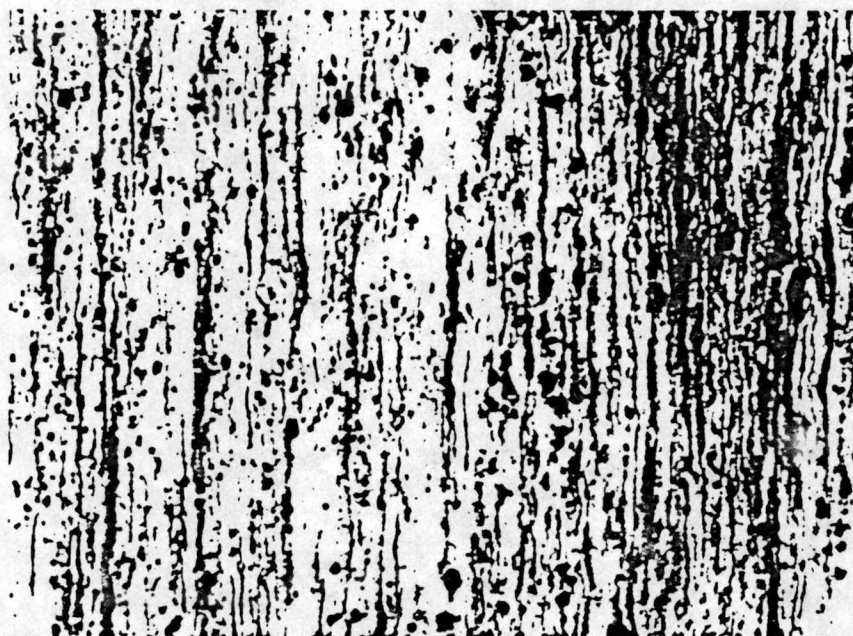


(b) Center

Fig. 34 Grain structure of the AR condition. 200X

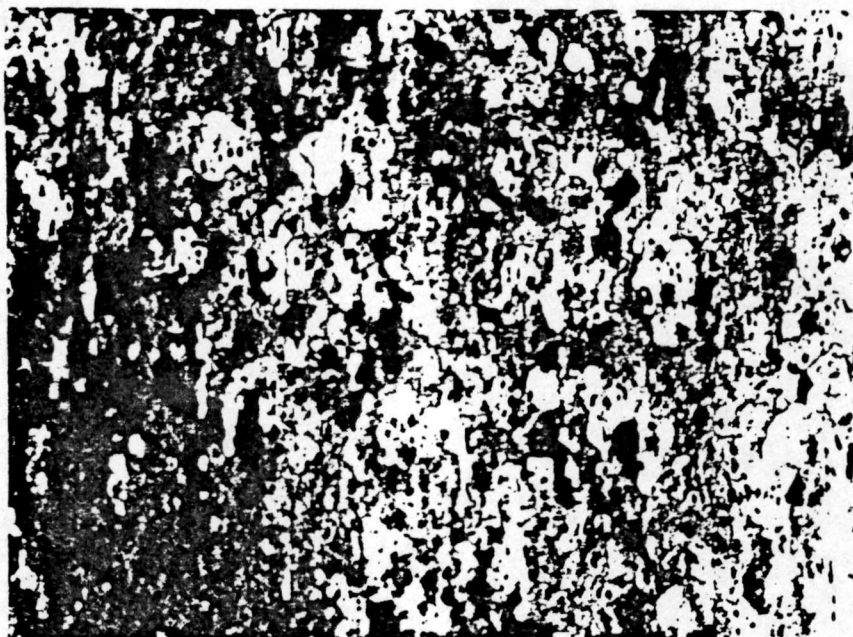


(a) 1-1 BA

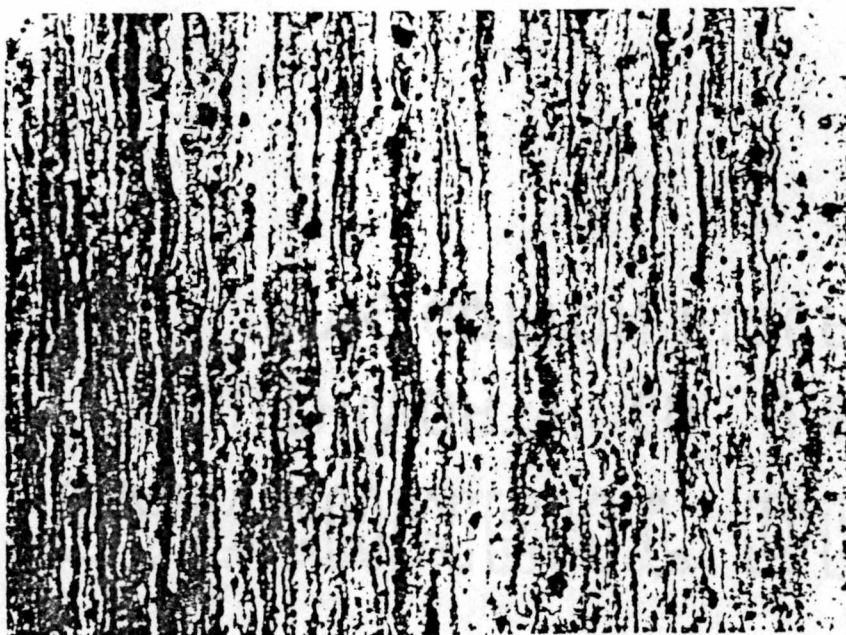


(b) 1-2 BA + CR88

Fig. 35 Grain structure of Prac. 1. 200X

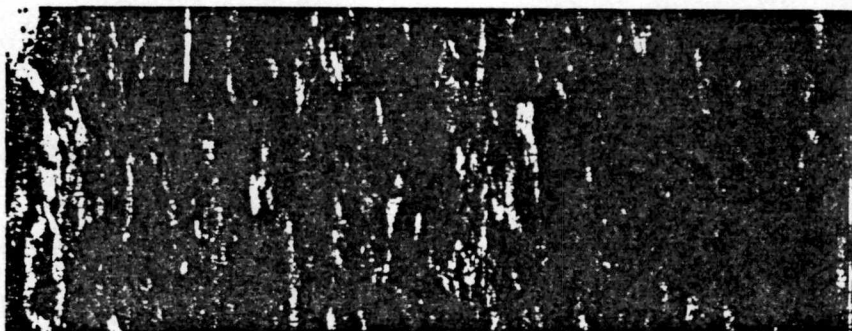


(a) 2-1 SA



(b) 2-2 SA + CR88

Fig. 36 Grain structure of Prac. 2. 200X



(a) 3-1 CR35

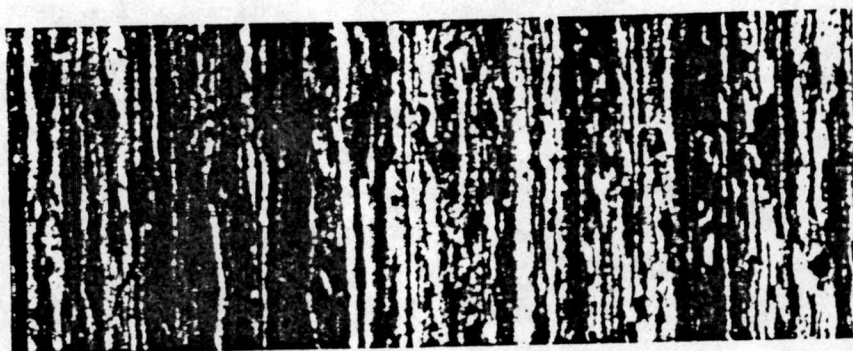


(b) 3-2 CR35 + BA



(c) 3-3 CR35 + BA + CR88

Fig. 37 Grain structure of Prac. 3. 200X



(a) 4-1 CR35



(b) 4-2 CR35 + SA



(c) 4-3 CR35 + SA + CR88

Fig. 38 Grain structure of Prac. 4. 200X

here are slightly larger than those in both BA and SA conditions in Prac. 1 and 2, comparing Fig. 37 (b) with Fig. 35 (a), and comparing Fig. 38 (b) with Fig. 36 (a), respectively.

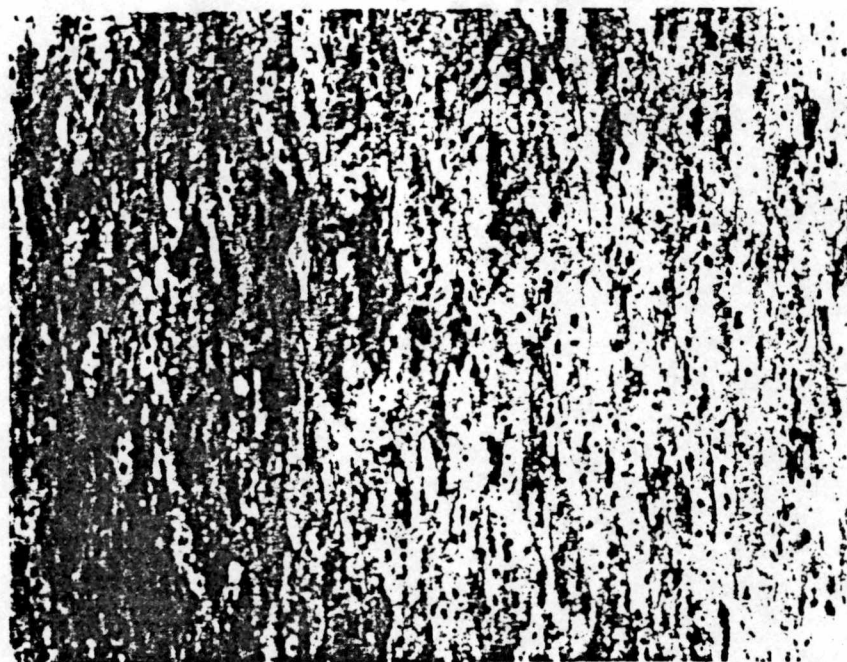
Fig. 39 shows the grain structure of Prac. 5. Compared with Prac. 3, Prac. 5 only had a prior BA from the AR condition, yet the intermediate BA did not affect the grain structure very much, seen in Fig. 37 (a) and Fig. 39 (b), the rest of the structures correspondingly showing the same results, Figs. 37 (b) and 39 (c), Figs. 37 (c) and 39 (d).

All the final CR88 states have the same microstructures, seen in Figs. 35 (b), 36 (b), 37 (c), 38 (c), and 39 (d), no matter whatever intermediate processing has been involved.

The primary and secondary particles, the distribution of size, density, and concentration play a very important role in the texture formation in the material [7, 9, 10, 12, 14, 15, 20, 24, 29, 32, 33, and 43]. Fig. 40 optically shows the primary particles, with and without using a special etching agent, 90 ml acetic acid, 5 ml of 1% HF and 5 ml of H_2SO_4 [33]. By using the special etchant, two kinds of primary particles were differentiated. The α type $\text{Al}_{15}(\text{Fe, Mn})_3\text{Si}_2$ etched dark and the $\text{Al}_6(\text{Fe, Mn})$ particles light [33], shown in Fig. 40 (b).



(a) 5-1 BA



(b) 5-2 BA + CR35

Fig. 39 Grain structure of Prac. 5. 200X



(c) 5-3 BA + CR35 +BA



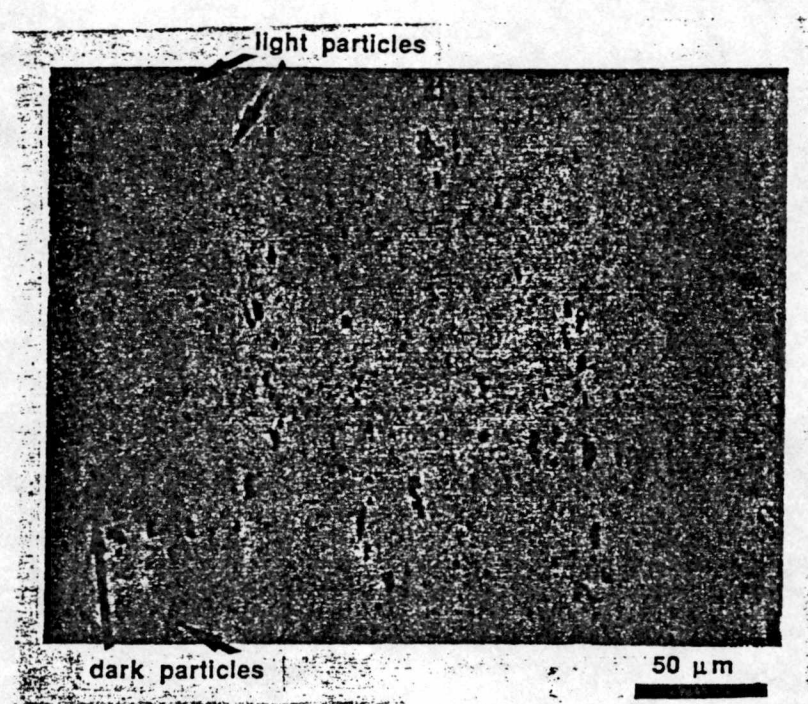
(d) 5-4 BA + CR35 + BA + CR88

Fig. 39 (continued).



(a) no etching, showing primary particles.

Fig. 40 Optical micrographs, showing primary particles.



(b) Specially etched, showing dark and light primary particles.

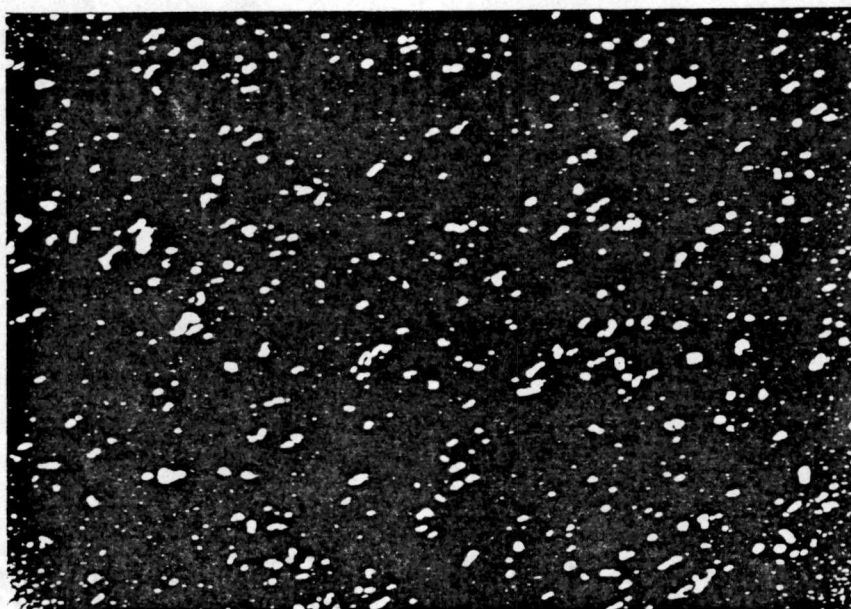
Fig. 40 (Continued)

SEM particle examinations were carried out in the research. Fig. 41 shows the microstructure of the AR condition material, revealing two general sizes of the particles. Figs. 42, 43 and 44 reflect the SEM observations of the two general sizes of particles in Pracs. 1 and 2 (Fig. 42), 3 and 4 (Fig. 43), and 5 (Fig. 44). Larger primary particles, which are blocky and slightly elongated along the rolling direction, are about 5 μm in size. The finer particles, sometimes called secondary particles [10, 23], are relatively equiaxed and about 0.5 μm or smaller in size. The primary particle sizes did not show very much change with the different processings.

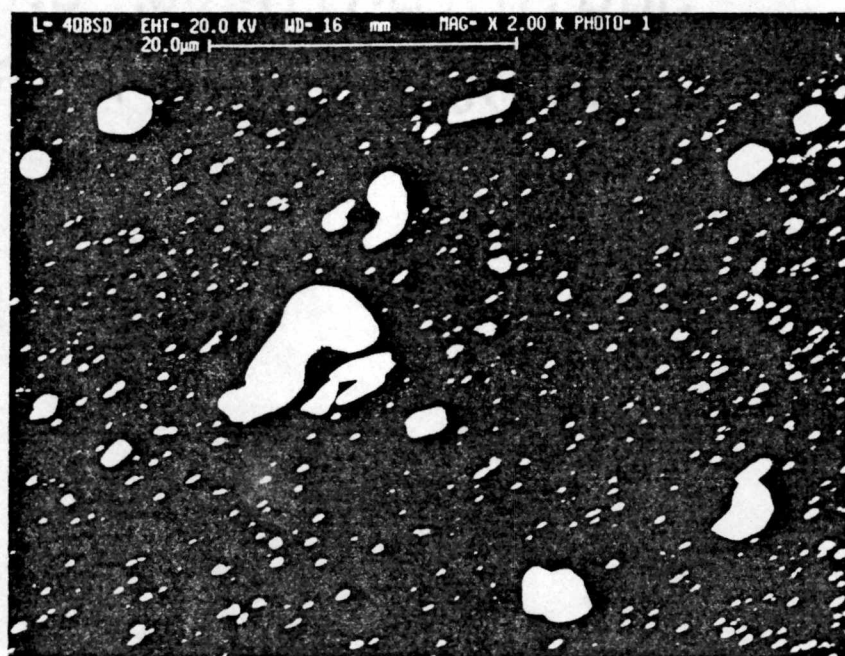
By image processing method (IPM) on the SEM, the primary particle percentage in average area was obtained for the 5 practices, tabulated in Table 6. The typical comparisons of the images before IPM and after IPM with the percentage readings are seen in Fig. 45. (a) is the original SEM image; (b), the IPM image processing in the same view, and (c) is the final calculation results by IPM.

By EDS chemistry analysis on the SEM, only the larger particles were analyzed, seen in Table 7. The chemical analyses in Table 7 indicate that three types of primary particles are labeled as shown. There was no obvious change in these after the various annealing steps.

The primary particle density per unit area (mm^2) for the 5 practices before the final CR88% was determined by manual measurement on the optical and SEM photographs. The results are seen in Table 8, and the values are similar to the range reported for 3004 alloy [33]. For all practices, except for practice 2, the values are

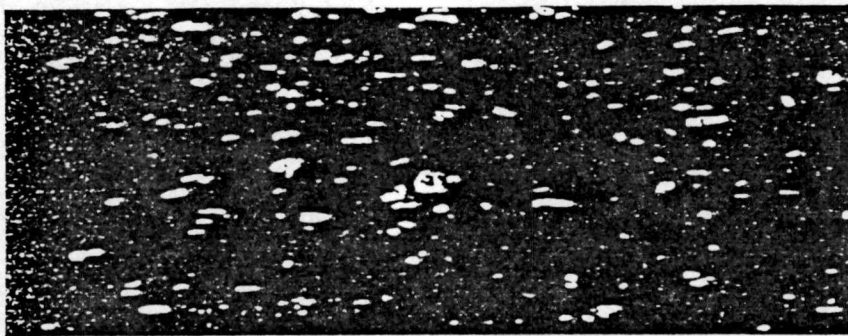


(a) 400X



(b) 2000X

Fig. 41 SEM micrographs (back - scattered electrons) of the material in the AR hot rolled condition. The specimen was unetched.



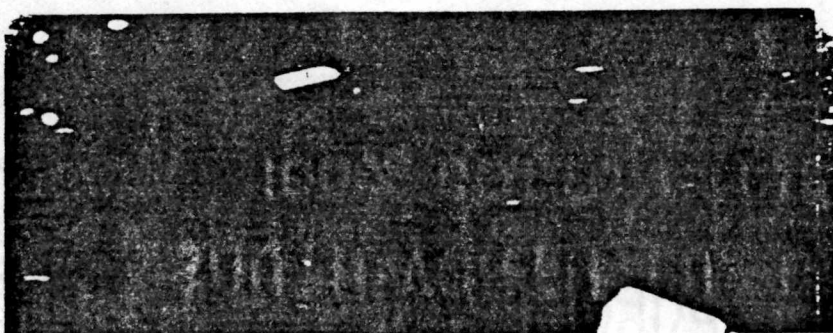
(a) 1-1 400X



(b) 1-1 5000X



(c) 2-1 400X

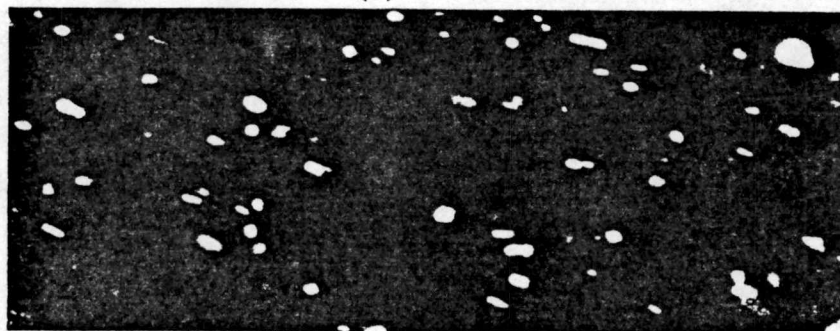


(d) 2-1 5000X

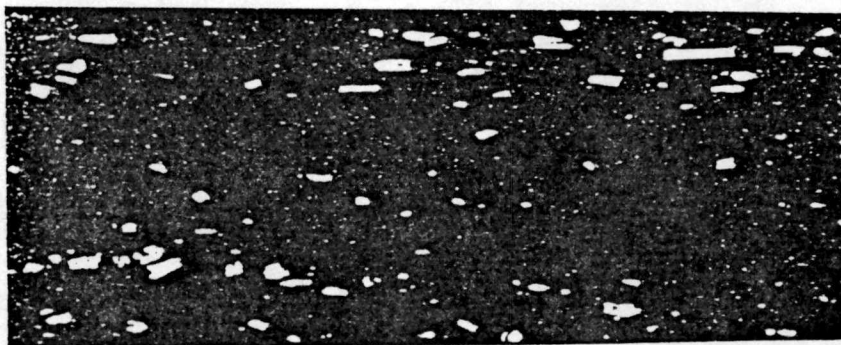
Fig. 42 SEM micrographs of the material in Pracs. 1 and 2 prior to the final CR88% state.



(a) 3-2 400X



(b) 3-2 5000X



(c) 4-2 400X

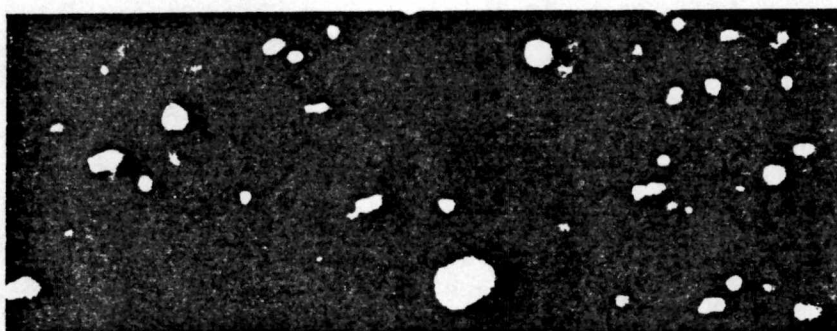


(d) 4-2 5000X

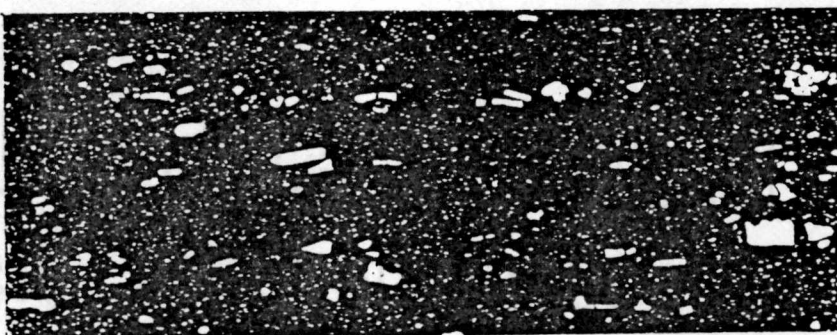
Fig. 43 SEM micrographs of the material in Pracs. 3 and 4 prior to the final CR88% state.



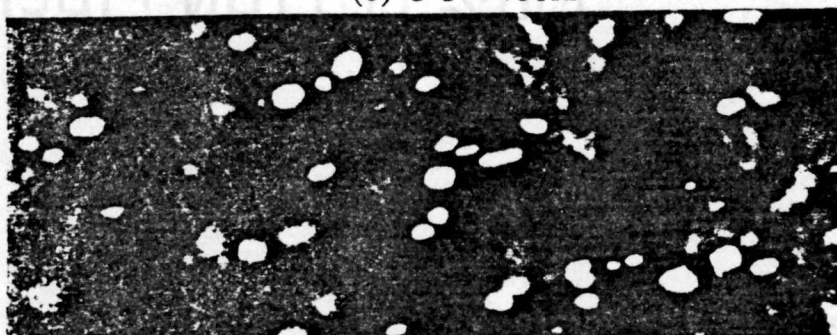
(a) 5-1 400X



(b) 5-1 5000X

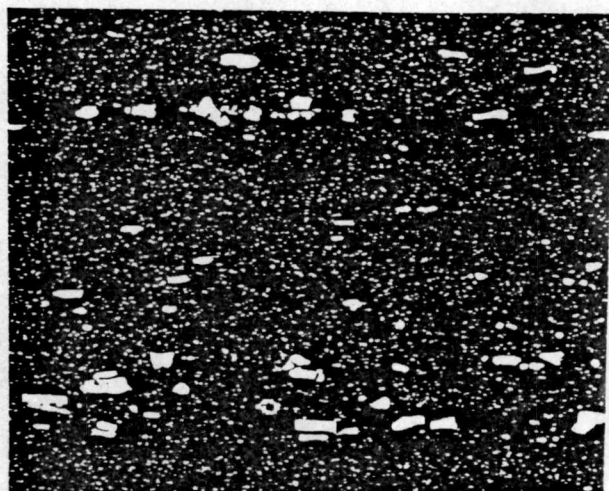


(c) 5-3 400X

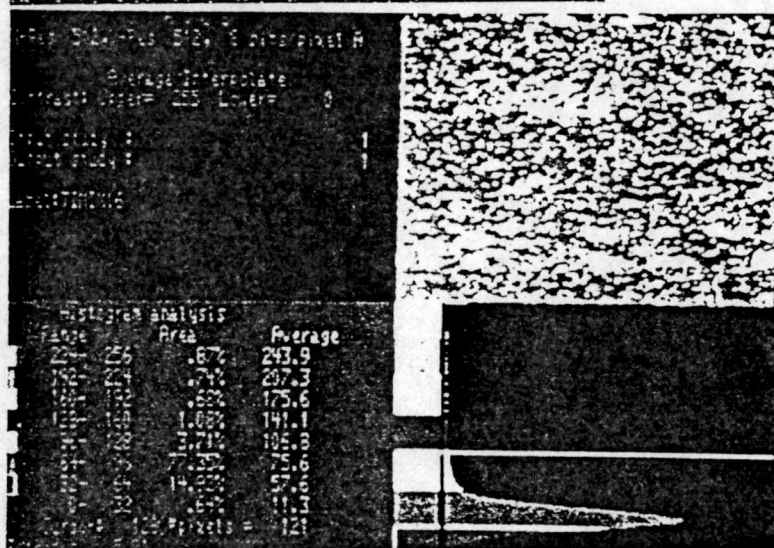


(d) 5-3 5000X

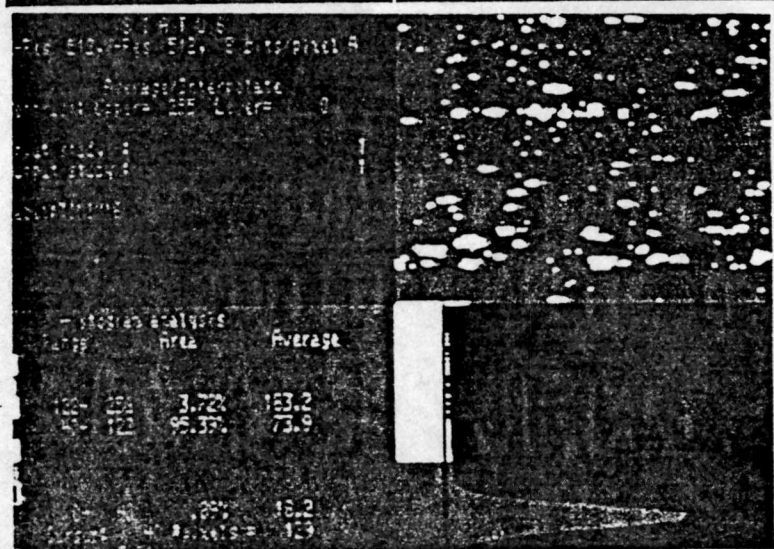
Fig. 44 SEM micrographs of the material in Pracs. 5 prior to the final CR88% state.



(a)



(b)



(c)

Fig. 45 Primary particle percentage in prac.3 before the final CR88% state by IPM.

Table 6 Particle percentage by IPM method.

Prac. #	Second. Particle (%)	Primary Particle (%)
As-received	2.7 ± 0.2	4.3 ± 0.4
1-1 (5-1)	2.5 ± 0.6	4.0 ± 0.0
2-1	1.9 ~	3.8 ~
3-2	1.9 ± 0.1	3.8 ± 0.0
4-2	3.0 ~	4.5 ~
5-3	3.1 ± 0.0	4.0 ± 0.5

Table 7 EDS microanalysis of primary particles (AR condition).

Phase	Composition atom % (wt%)					
	Fe	Mn	Mg	Si	Cu	Fe/Mn
α - Al ₁₅ (Fe, Mn) ₃ Si ₂	2 (5)	4 (7)	0.9 (0.8)	0.1 (0.1)	0.2 (0.3)	0.5 (0.7)
	1 (2)	2 (3)	0.9 (0.8)	0.3 (0.3)	0.1 (0.1)	0.5 (0.7)
Al ₆ (Fe, Mn)	10 (17)	8 (14)	0.5 (0.3)	3.9 (3.4)	0.3 (0.7)	1.3 (1.2)
	9 (15)	9 (16)	0.1 (0.1)	4.2 (3.7)	0.3 (0.7)	1.0 (0.9)
	7 (13)	6 (11)	0.5 (0.4)	0.2 (0.2)	0.3 (0.7)	1.1 (1.2)
	6 (10)	7 (12)	0.4 (0.3)	3.3 (3.0)	0.2 (0.4)	0.9 (0.8)
	5 (10)	6 (11)	0.6 (0.5)	2.2 (2.1)	0.1 (0.2)	0.8 (0.9)
	8 (13)	11 (18)	0.1 (0.1)	4.4 (3.8)	0.2 (0.4)	0.7 (0.7)

Table 8 Primary particle density for the annealed conditions of the five practices.

Practice	Particle density (number/ mm ²) [± 100]
As-received (hot rolled)	700
Prac. 1: hot rolled + batch annealed	700
Prac. 2: hot rolled + strip annealed	500
Prac. 3: hot rolled + + cold rolled 35% + batch annealed	600
Prac. 4: hot rolled + + cold rolled 35% + strip annealed	600
Prac. 5: hot rolled + batch annealed + cold rolled 35% + batch annealed	700

similar to that of the AR condition. Thus, they do not provide any clear indication of the causes of the effect of processing practice on the earing behavior.

Practice 2 had lower particle density, consistent with the SEM observations. And IPM also gave a lower reading* for practice 2. Therefore, the lower earing for practice 2 seems to be connected with the lower particle density, lower primary particle percentage.

It appears, then, that the factors governing the effect of processing practice on earing behavior reside in the fine microstructural features, such as the relatively fine secondary particles or finer (unresolved) particles, the annealed dislocation cell structure, and the matrix solute content (which is related to the amount and types of precipitates present), for which we have to turn to transmission electron microscopy (TEM). It is clear that the intermediate 35% cold rolling and subsequent annealing is the processing factor which caused the higher earing, and this suggests that the deformation structure after the cold rolling determines the subsequent annealing texture which then, in turn, affects the final 88% cold rolling texture, and causes the high degree of earing.

* The reading was from one set of measurements, but the observations were evident that practice 2 had lower density.

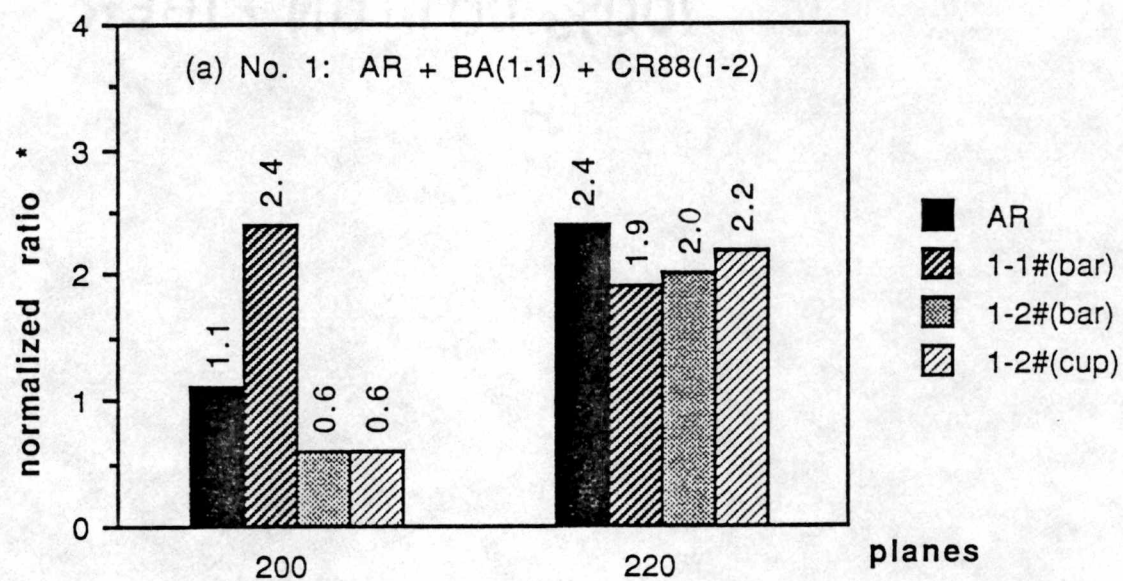
IV. 3 Texture Detection and Analysis

X-ray diffraction intensity ratios for {200}, {220} and {422} planes are plotted in Figs. 46, 47, 48, 49, and 50 respectively. The reproducibility of the ratios was within ± 0.2 .

As-received (AR) condition for {200}, {220} and {422} plane x-ray diffraction intensity is plotted in Figs. 46-49 respectively, shown in black (or white in Fig. 49) column. Samples from both tensile bar and earing cup were tested and the results are reflected in every figure, shown as "bar" and "cup".

Prac. 1 and 2 gave identical x-ray diffraction intensities, see {200} and {220} peak ratios in Fig. 46 (a) with (b). {200} peak ratios increased substantially from 1.1 (AR condition) to 2.4-2.5 after batch anneal (BA) and strip anneal (SA), yet dropped to 0.6-0.7 after final CR88 in both cases, which reflects the cube texture variation tendency. {220} peak ratios, however, did not show very much change, ranging from 2.4 to 1.7, which may imply that from AR condition to final different treatment, the Goss and brass textures have kept in a relatively high level and changed a little. Results from tensile bars and from earing cups were identical, which may imply that during the earing test no further cube, Goss and brass textures evolved.

Prac. 3 and 4 gave different x-ray diffraction intensities, see {200} and {220} peak ratios in Fig. 47 (a) and (b). {200} peak ratios of 3-1 (CR35) should have had the same value as that of 4-1 (CR35), but were 1.8 for 3-1 and 0.8 for 4-1. This difference may seem to have something to do with the AR condition, especially the hot rolled



* peak integral ratio of sample/standard powder sample

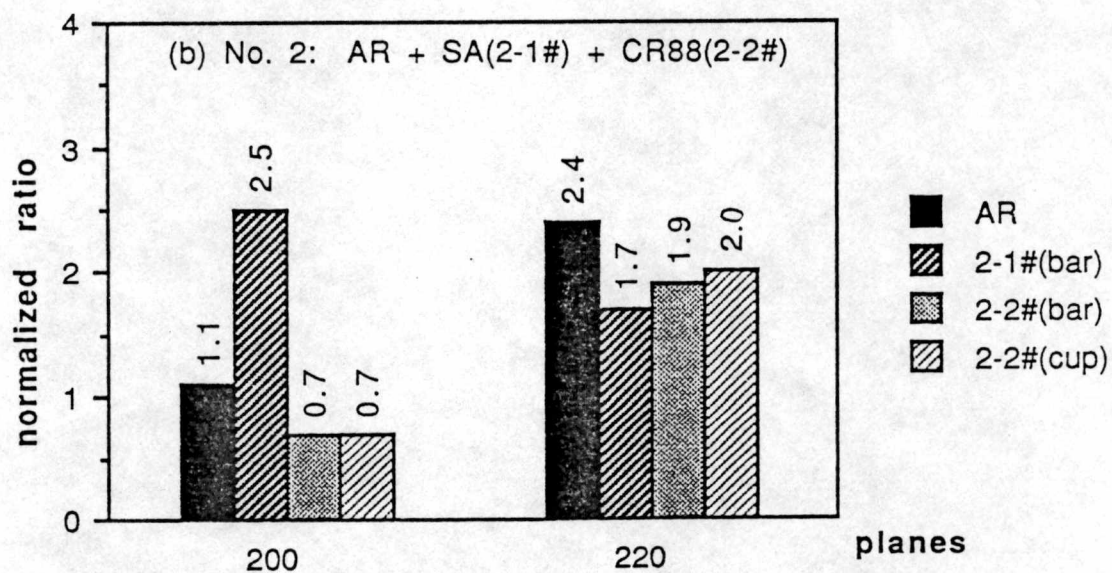


Fig. 46 {200}, {220} plane x-ray diffraction for Pracs. 1 and 2.

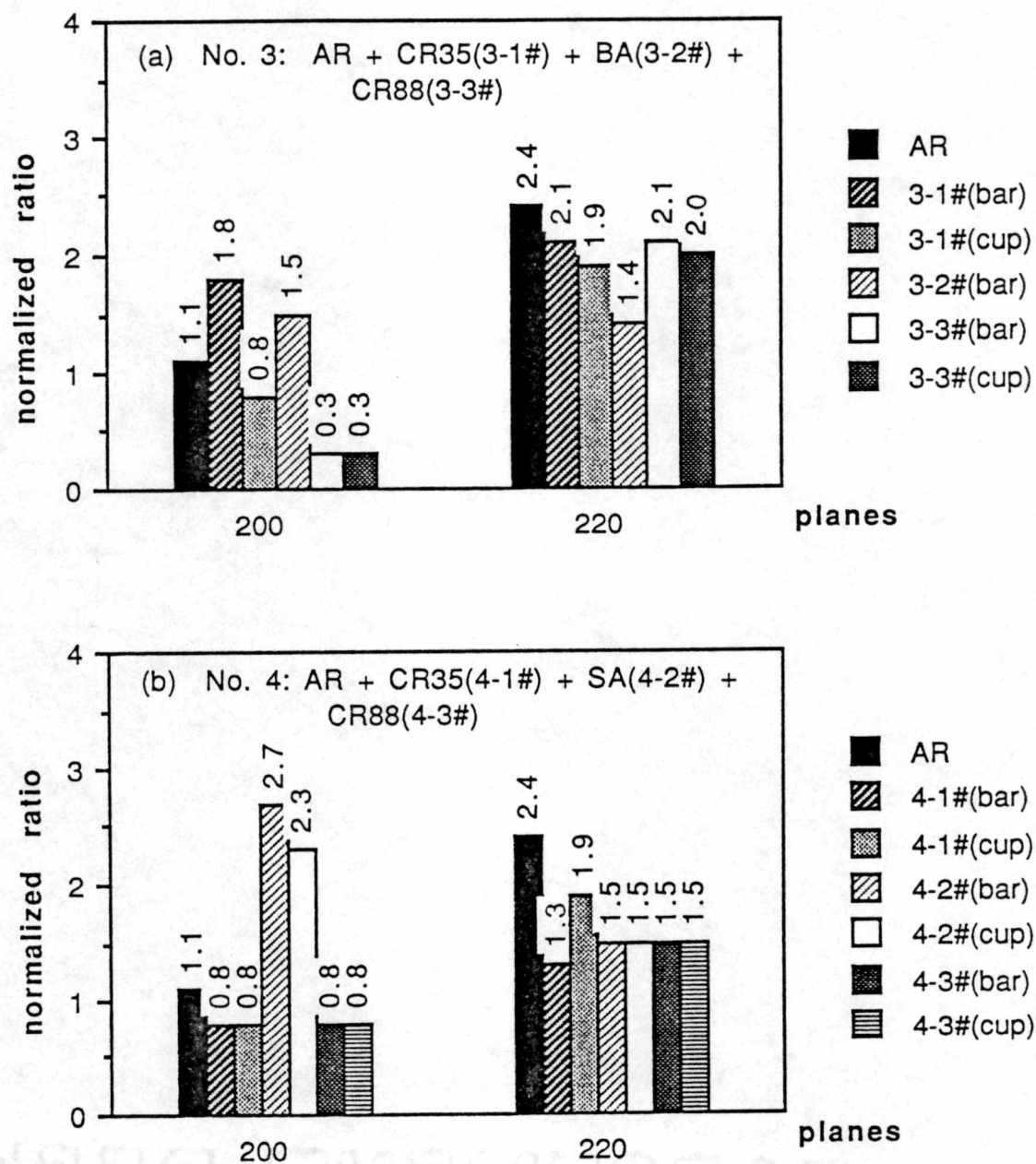


Fig. 47 {200}, {220} plane x-ray diffraction for Pracs. 3 and 4.

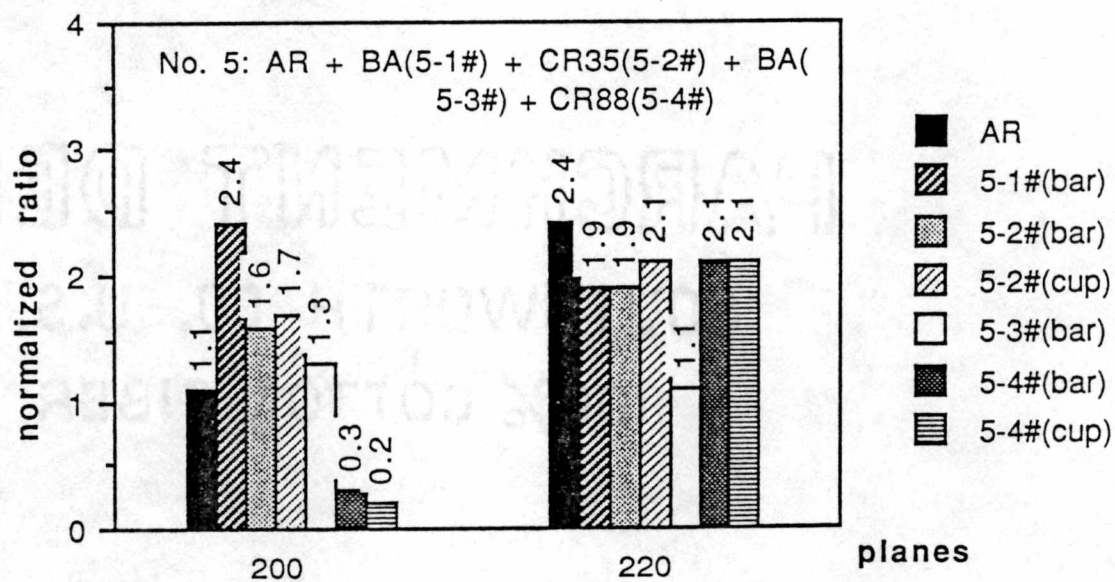


Fig. 48 {200}, {220} plane x-ray diffraction for Prac. 5.

422 peak ratio after cr88 of all practices

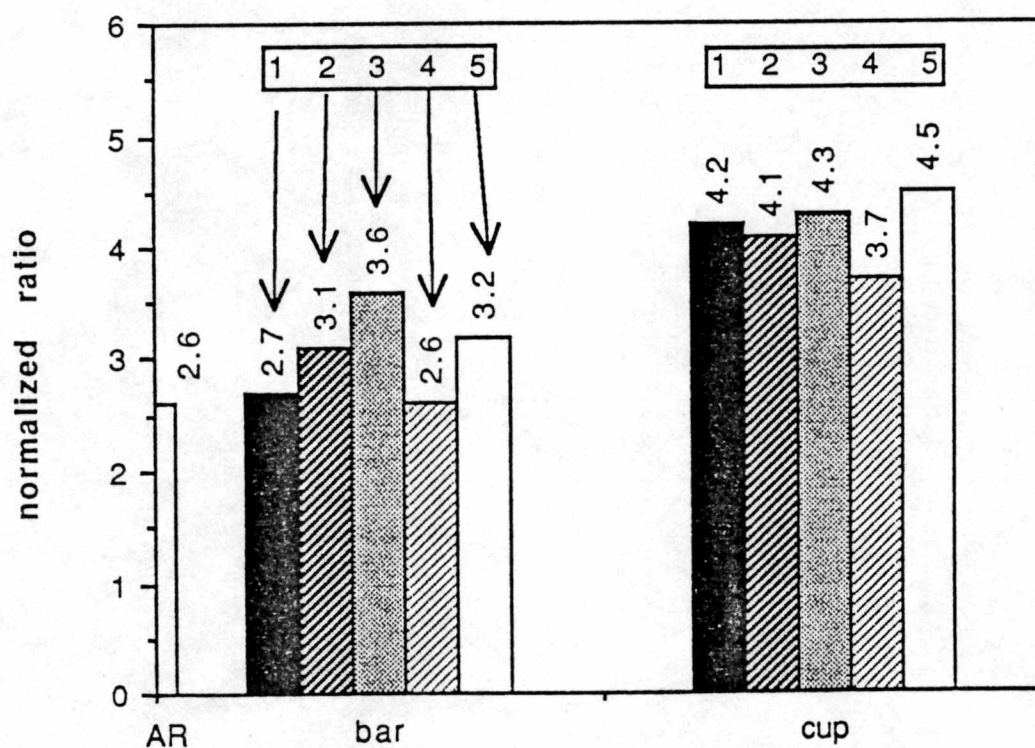


Fig. 49 {422} plane x-ray diffraction ratio after CR88 of all practices, AR condition is plotted on the left.

200, 220, 422 ratios after final CR88 conditions

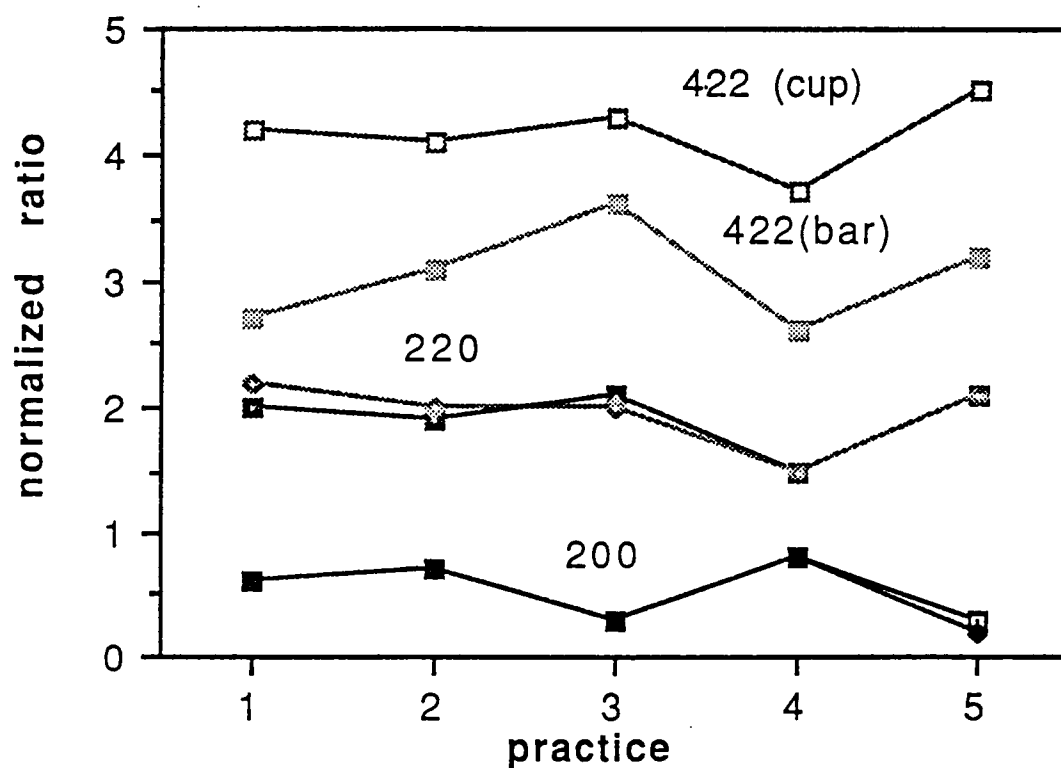


Fig. 50 {200}, {220}, and {422} plane x-ray diffractions for all the final CR88% condition.

reduction, seen in Fig. 37 (a) and Fig. 38 (a). Fig. 38 (a) showed more reduced structure, therefore gave lower $\{200\}$ peak ratio. Results from tensile bars and from earing cups were identical, which may also imply that during the earing test no further cube, Goss and brass textures evolved.

Fig. 48 shows $\{200\}$ and $\{220\}$ plane intensities for Prac. 5. Compared with Prac. 3 (seen in Fig. 47 (a)), 5-1 (BA) did not significantly change the $\{200\}$, $\{220\}$ peak ratios in different individual procedures, see the results from 5-2 and 3-1 (for $\{200\}$ peak ratios, 1.6 from 5-2 and 1.8 from 3-1; for $\{220\}$ peak ratios, 1.9 from 5-2 and 2.1 from 3-1), from 5-3 and 3-2 (for $\{200\}$ peak ratios, 1.3 from 5-3 and 1.5 from 3-2; for $\{220\}$ peak ratios, 1.1 from 5-3 and 1.4 from 3-2), from 5-4 and 3-3 (for $\{200\}$ peak ratios, 0.3 from 5-4 and 0.3 from 3-3; for $\{220\}$ peak ratios, 2.1 from 5-4 and 2.1 from 3-3).

Fig. 49 shows $\{422\}$ plane x-ray diffraction intensities after CR88 for all practices; AR condition is plotted on the left. Although $\{422\}$ plane peak ratios for all the conditions have kept at a very high level and showed only a little change, which may imply that Cu type texture has kept in the same level throughout all the practices, and the pole figure results will show this phenomenon too. The intensity from the cup samples was higher than that from the tensile samples, but the reason was not determined.

Fig. 50 shows $\{200\}$, $\{220\}$, and $\{422\}$ plane x-ray diffraction intensities for all the final CR88% condition. Using the ratio "r" of $\{200\}$ plane intensity over $\{220\}$ plane intensity is used to predict

earing, Precht et al. [58] found out that there was an inverse proportionality between earing percentage and r for Al alloy 3004 after H19 treatment. If a subscript here means the practice number, then $r_1 = 0.6/2.2 = 0.27$, $r_2 = 0.7/2 = 0.35$, $r_3 = 0.3/2 = 0.15$, $r_4 = 0.8/1.5 = 0.53$, and $r_5 = 0.3/2.1 = 0.14$. Except r_4 , all of them fall into the category of Precht et al.'s work [58]. Thus the r ratio can be a dependable tool to show the earing strength for the final cold rolled condition.

It has been a controversial issue whether the surface of rolled plate or the center of it should be checked to detect the texture as a standard [59]. As a result, the intensity variations throughout the thickness were detected for Prac. 3-1 (CR35), which had a thickness of 3.30 mm (0.130 in.). The results showed that both {200} and {220} plane intensities varied, seen in Table 9. (The multi-layer etch off procedure is shown in the sketch at the bottom of the table.) The maximum value of r , the ratio of {200} plane intensity over {220} plane intensity, was 0.508 mm (0.02 in.) beneath the surface*. However, the extensive 88% reduction in thickness should make the texture uniform, so the x-ray diffraction results from the surface should indicate the proper texture.

* The surface here means the rolled surface after thoroughly cleaning.

Table 9 X-ray diffraction results of Prac. 3-1 in multi-layer etch off method

Prac. #	Thick. mm (in)	Ratio (200)	Ratio (220)	R (200)/R (220)
3-1 (1)	3.39 (0.130)	1.8	2.1	0.9
3-1 (2)	3.15 (0.124)	2.4	0.8	3.0
3-1 (3)	2.79 (0.110)	3.2	0.5	6.4
3-1 (4)	2.34 (0.092)	2.8	0.7	4.0
3-1 (5)	1.98 (0.078)	2.4	1.0	2.4
3-1 (6)	1.78 (0.070)	2.6	1.0	2.6

{111} pole figures for the AR and all the final CR88% conditions are seen in Figs. 51 (AR condition), 52 (Prac. 1), 53 (Prac. 2), 54 (Prac. 3), 55 (Prac. 4), and 56 (Prac. 5). {200} pole figure for Prac. 2 after final CR88% condition is also shown in Fig. 57, comparing with Fig. 53.

Comparing Fig. 51 with Figs. 52-56, it is obvious that the texture for the AR condition is similar to the cold rolled texture, which is a copper type*. This is because the exit temperature after hot rolling was between 304 and 316 °C, well below the 500 °C critical one, below which only the copper type texture exists in terms of pole figure [43]. However, one of the differences between AR condition and the CR88 state was that the maximum intensity contour values of cube texture for AR condition were higher (2.6 ~ 2.9) than those of the CR88 state (1.6 ~ 1.9), which means that AR condition had higher cube texture component. The x-ray diffraction results also reflected this phenomenon (Figs. 46 - 48).

Pole figures in Figs. 52-56 show a similar copper type texture, which now stands for at least 4 kinds of textures, brass type {110}<112>, pure copper type {112}<111>, Goss type {110}<001>, and S type {123}<634>, represented respectively as "@", "#", "\$", and "&". The cube component was signified by "*".

* Note: I assume that copper type and pure copper type are different; the former one is a complex one of {112} <111>, {110}<112>, {110}<001> (or Goss type), even {100}<001> cube one and other minor ones, the latter one is only pure {112}<111> type.

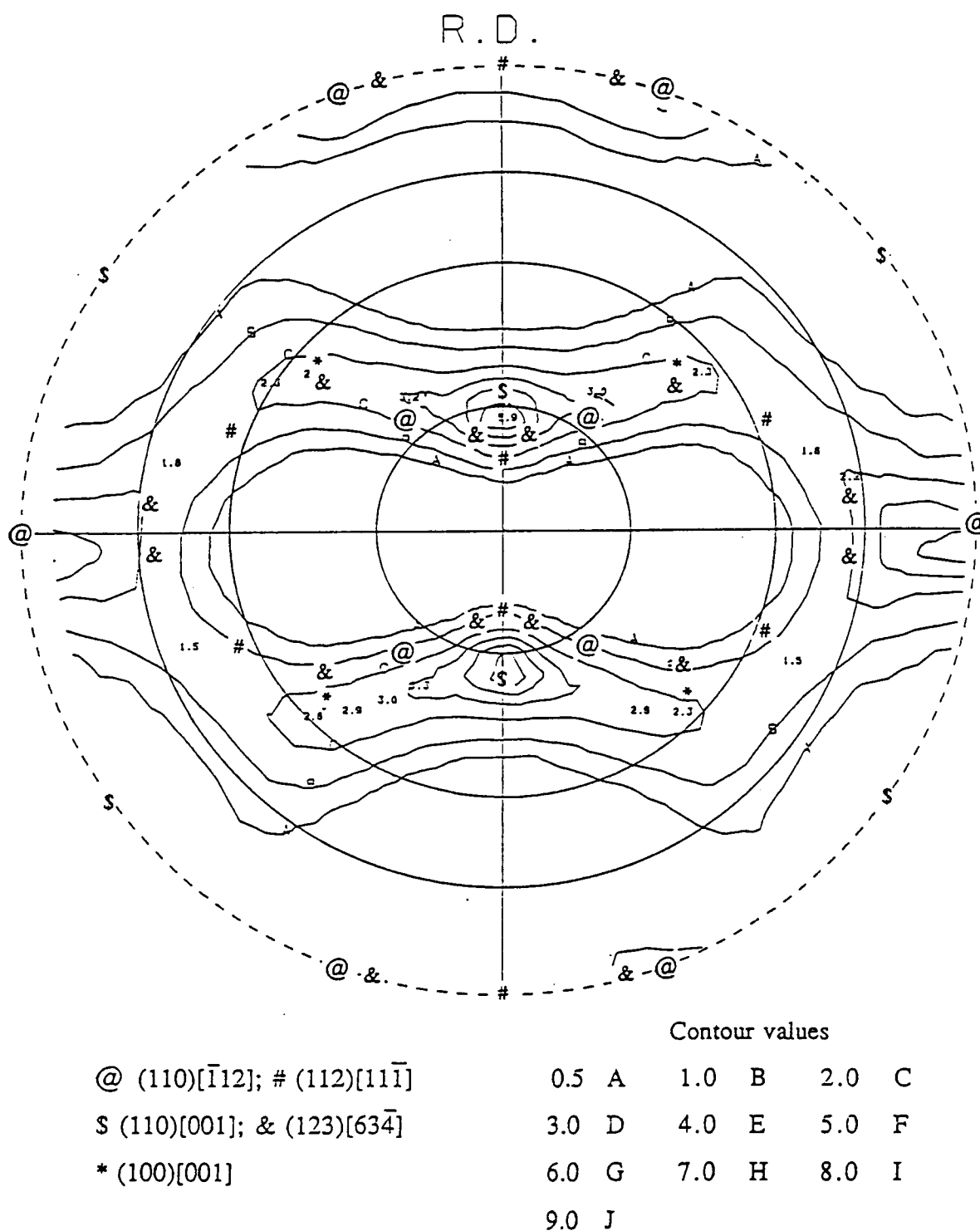
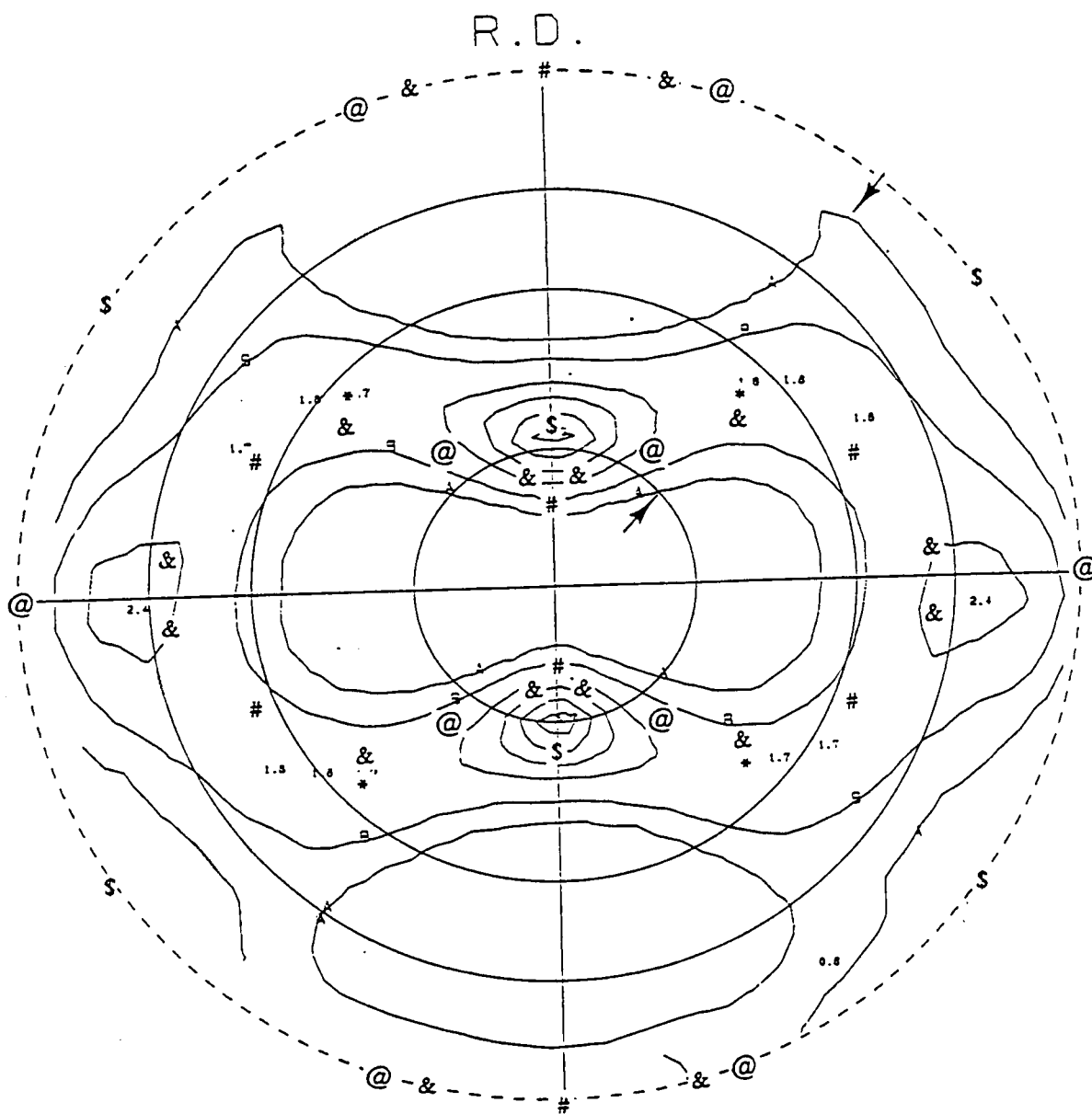


Fig. 51 {111} pole figure for AR condition.



Contour values			
@ (110)[$\bar{1}12$]; # (112)[$11\bar{1}$]	0.5 A	1.0 B	2.0 C
S (110)[001]; & (123)[$63\bar{4}$]	3.0 D	4.0 E	5.0 F
* (100)[001]	6.0 G	7.0 H	8.0 I
	9.0 J		

Fig. 52 {111} pole figure for Prac. 1 in final CR88% condition.

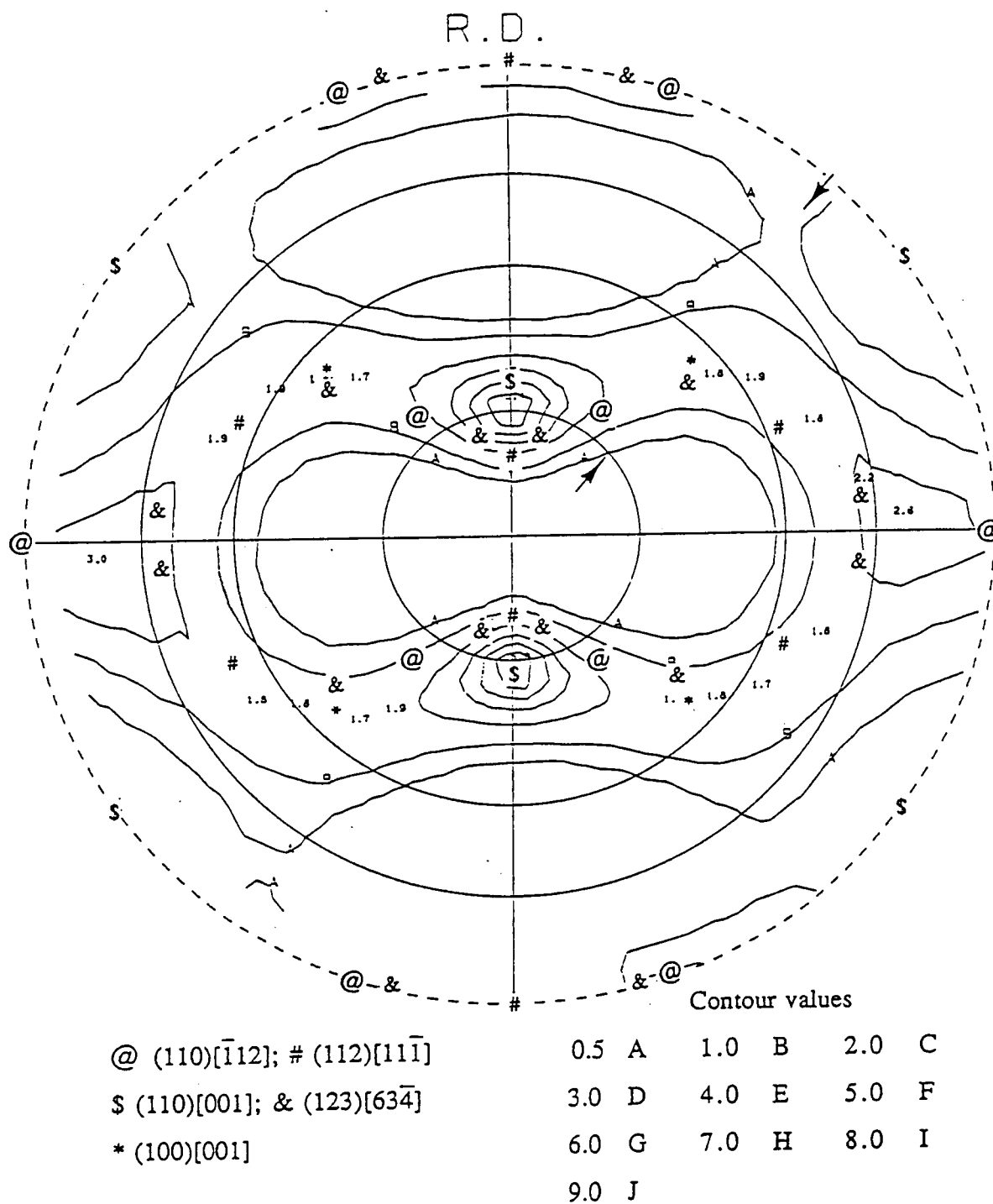


Fig. 53 {111} pole figure for Prac. 2 in final CR88% condition.

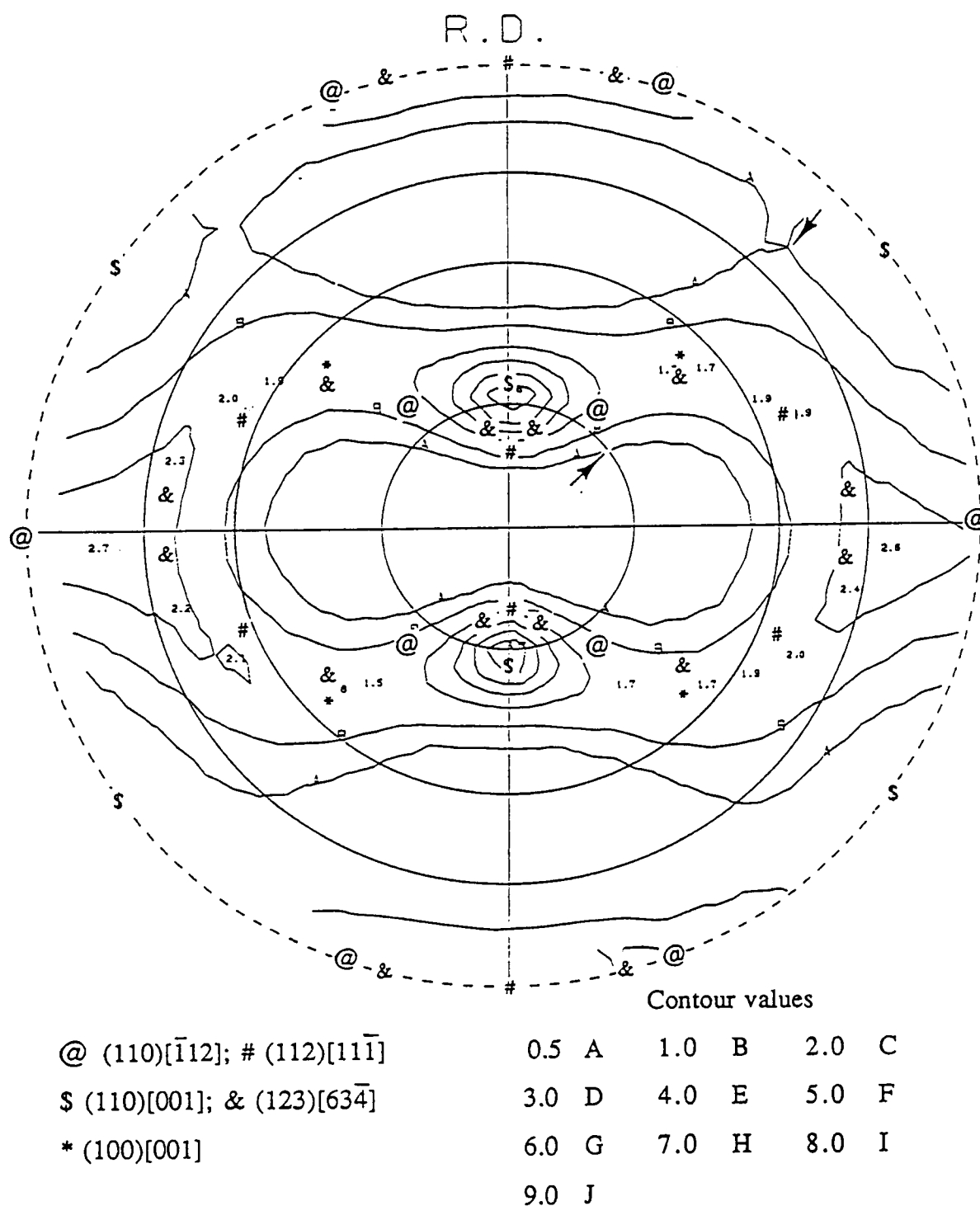
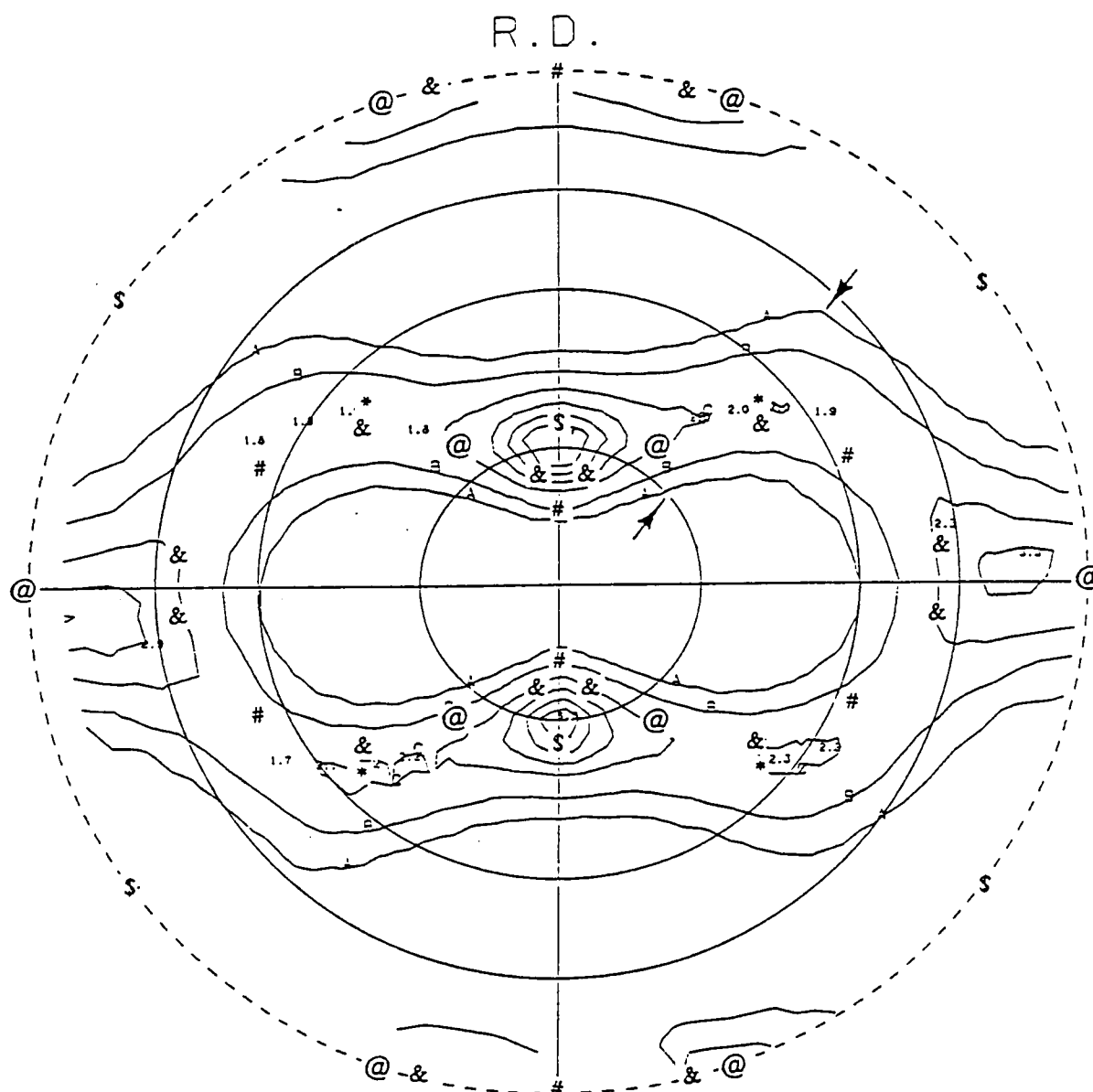
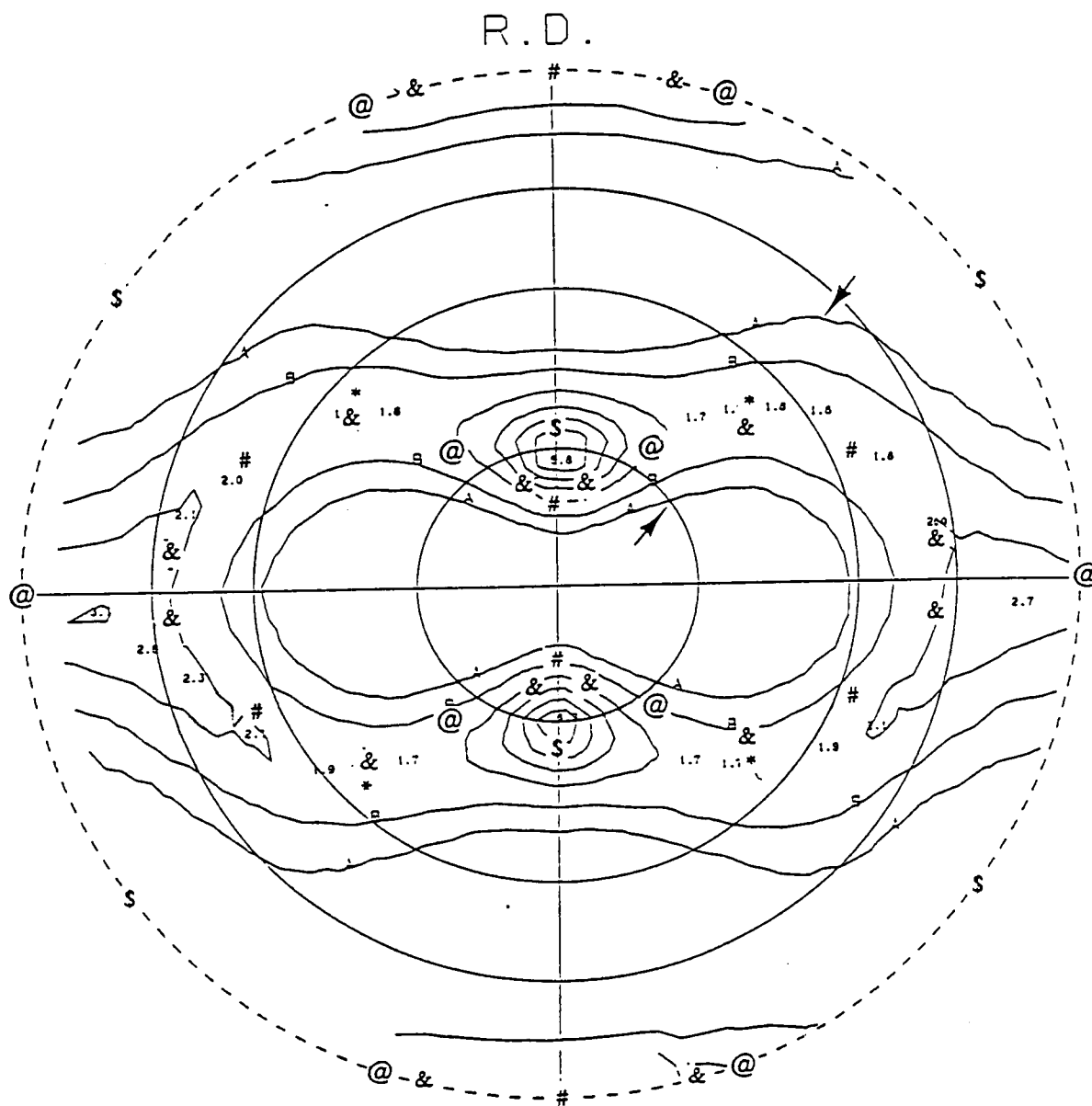


Fig. 54 {111} pole figure for Prac. 3 in final CR88% condition.



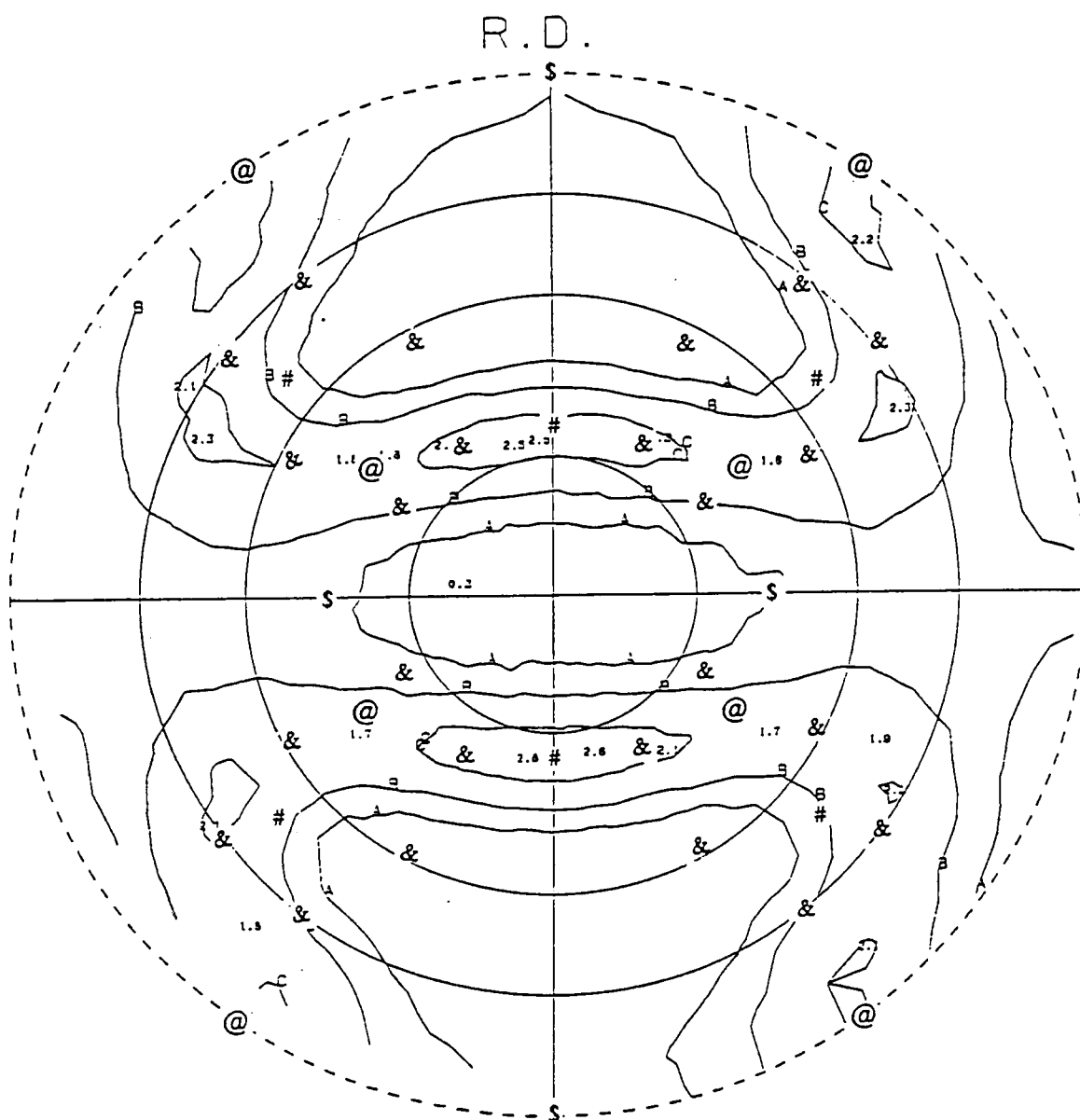
Contour values				
@ (110)[$\bar{1}12$]; # (112)[$1\bar{1}\bar{1}$]	0.5 A	1.0 B	2.0 C	
\$ (110)[001]; & (123)[$63\bar{4}$]	3.0 D	4.0 E	5.0 F	
* (100)[001]	6.0 G	7.0 H	8.0 I	
	9.0 J			

Fig. 55 {111} pole figure for Prac. 4 in final CR88% condition.



Contour values						
@ (110)[$\bar{1}12$]; # (112)[$1\bar{1}\bar{1}$]	0.5	A	1.0	B	2.0	C
\$ (110)[001]; & (123)[$6\bar{3}4$]	3.0	D	4.0	E	5.0	F
* (100)[001]	6.0	G	7.0	H	8.0	I
	9.0	J				

Fig. 56 {111} pole figure for Prac. 5 in final CR88% condition.



	Contour values					
@ (110)[$\bar{1}12$]; # (112)[$11\bar{1}$]	0.5	A	1.0	B	2.0	C
\$ (110)[001]; & (123)[$63\bar{4}$]	3.0	D	4.0	E	5.0	F
* (100)[001]	6.0	G	7.0	H	8.0	I
	9.0	J				

Fig. 57 {200} pole figure for Prac. 2 in final CR88% condition, comparing with Fig. 52.

From $\{111\}$ pole figures mentioned above, Figs. 52-56, it seems true that cube texture $\{100\}\langle 001 \rangle$ might have played a significant role in the final cold rolled complex texture (cube texture maximum intensity contour values were about $1.6 \sim 1.9$) in the texture of the final CR88 states of the five practices. However, from $\{200\}$ pole figure (Fig. 57), it was clear that the cube texture $\{100\}\langle 001 \rangle$ did not play an important role in the final complex texture, which is consistent with the results from the x-ray diffraction experiment (Figs. 46 - 48, $\{200\}$ intensities in the final states).

The difference in the rolling textures, from these pole figures, can be visually identified by observing the spread around orientations rotated away from cube texture and around the rolling texture components [17] (arrowed in Figs. 52-56).

Table 10 shows the maximum intensities in the $\{111\}$ pole figures. Maximum intensities in these $\{111\}$ pole figures (Figs. 52-56) showed that Pracs. 3, 4, and 5 had higher values than Pracs. 1 and 2, which are consistent with the earing results. And the maximum intensity region corresponded with Goss texture $\{110\}\langle 001 \rangle$, S texture $\{123\}\langle 634 \rangle$, and pure copper texture $\{112\}\langle 111 \rangle$, which means that these three texture components should have higher contributions. X-ray diffractions from $\{220\}$ and $\{422\}$ planes did not clearly show this tendency, and unfortunately $\{123\}$ plane intensity from the x-ray diffraction could not be checked due the limitation of the technique.

Table 10 Maximum intensities in {111} pole figures from the five
final CR88 states.

Prac.#	1	2	3	4	5
Upper	5.3	5.4	5.6	5.9	5.8
Lower	5.7	5.9	6.7	6.5	6.9
Average	5.6	5.7	6.2	6.3	6.4

R values were measured from the tensile test samples for all the 5 practices. The data can be seen in Appendix 4. R value can be related to earing strengths in some alloys. The R values obtained here are shown as the plot from Wilson and Butler [37], in Fig. 58. My results are represented as I: 1-2; J: 2-2; K: 3-3; M: 4-1, 3-1; N: 4-2; O: 4-3; Q: 5-2; T: 5-4. They agreed satisfactorily with those of Wilson et al.'s results.

Only the 88% rolled samples were examined in this technique and the data are plotted in Table 11, which shows the consistency of both earing strength and earing position with those of earing tests.

IV. 4 Closure

The post-treatment investigations for all of the five practices are focused on the relationship of earing occurrence and strength (EOS) with individual thermal and mechanical processing in terms of tensile property directionality (TPD), particle characteristics, and etc. EOS was examined by the experiment of earing cup test, of x-ray diffraction intensity ratios for {200}, {220}, and {422} planes, of pole figure for {111} and {200} planes, of R values, and of ultrasonic technique. There was no clear correlation between the TPD and the EOS, nor was there one between primary particle characteristics and EOS. However, EOS correlated with ultrasonic technique and R value results satisfactorily. Both x-ray diffraction intensity ratio and pole figure results showed the major texture components which existed in the final cold rolled conditions. Those texture components are believed to be responsible for the EOS.

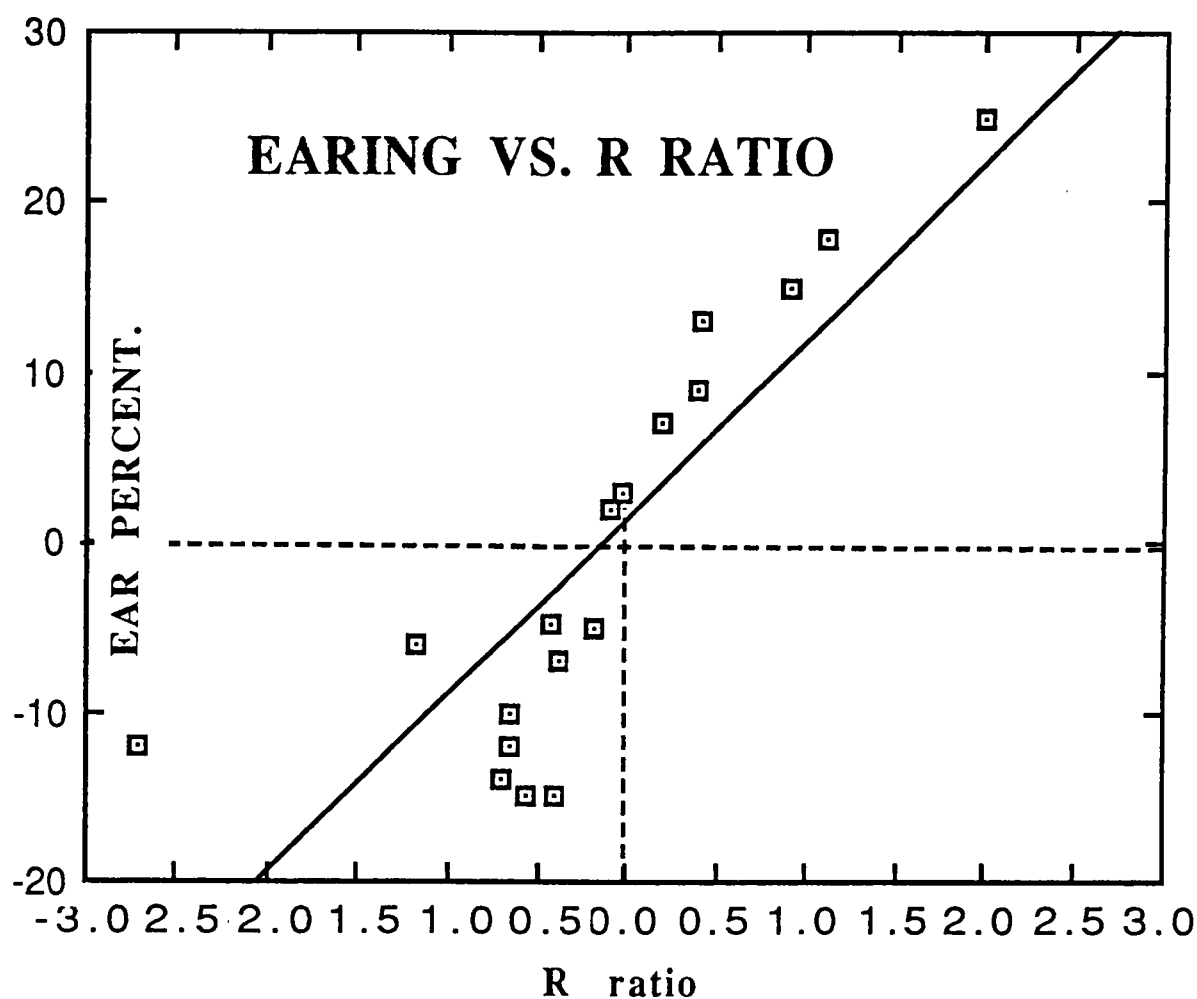


Fig. 58 The relation between the mean earing percentage and the ratio of $(R_0, 90-R_{45})/R_a$ [37],

where $R_a = (R_0 + R_{90} + 2 \times R_{45})/4$, where $R_{0,90} = (R_0 + R_{90})/2$

My results:

I: 1-2; J: 2-2; K: 3-3; M: 4-1, 3-1;

N: 4-2; Q: 4-3; Q: 5-2; T: 5-4

Table 11 Ultrasonic test results* from final CR88% condition of all 5 practices.

No.	$-\rho_{3/1000}^{**}$	Degree***
1-2 (BA+CR88)	-3.6	45
2-2 (SA+CR88)	-3.2	45
3-3 (CR35+BA+CR88)	-5.3	45
4-3 (CR35+SA+CR88)	-5.2	45
5-4 (BA+CR35+BA+CR88)	-5.8	45

* These tests were conducted by Dr. Lu and his colleagues in the Engineering Mechanics Department, University of Kentucky, Lexington, KY 40506.

** $-\rho_{3/1000}$ is a special symbol based on the arguments of Dr. Lu. It represents the earing strength; the more negative it is, the higher the earing strength is.

*** The degree shown here means the oriented degree of earing to the RD.

The processing involved with intermediate CR35 and annealing gave a higher earing strength, and this correlates with the change in the maximum intensities in the {111} pole figures.

CONCLUSIONS AND PROJECTIONS

V. 1. Conclusions

1. The tensile mechanical properties in the sheet material were not dependent on the direction of measurement in all the conditions.
2. The three processing practices that involved an intermediate 35% cold rolling produced slightly lower strengths and higher ductilities in the final 88% cold rolled material.
3. The faster heating rate to the annealing temperature, which simulated that associated with strip annealing, gave a finer annealed grain size than did the slower heating rate, which approximated that for batch annealing. However, where the heating rate was the only variable, no effect on earing was obtained.
4. The primary particle density in the annealed condition of the five practices did not correlate with the differences in earing found.
5. The results show that the processing factors causing the differences in earing are the intermediate 35% cold rolling and the effect of the subsequent annealing prior to the final 88% cold rolling.
6. X-ray diffraction data showed that $\{220\}$ and $\{422\}$ plane intensities in all final 88% cold rolled condition ranged from 2-4 times over that of the random sample, and both x-ray

diffraction and pole figure results showed that the {200} plane intensity did not contribute very much to the final cold rolled texture. {220} x-ray intensities were found to correspond with {110}<112> and {110}<001>, and {422} ones with {112}<111> rolling textures in the pole figures. The pole figures showed that {110}<112>, {110}<001>, {112}<111>, and {123}<634> components played very important roles in the complex texture problem, especially in practices 3, 4 and 5; however, the x-ray diffraction results did not reflect this tendency.

7. X-ray diffraction data from as-received condition showed that {220} and {422} plane intensities were about 2.5 times that of the random sample. Both x-ray diffraction and pole figure results showed that the as-received condition had a complex texture similar to the cold rolled texture. These, along with the microstructural observations, proved that the AR condition was not a hot rolled condition, rather a "warm" rolled condition, which means that the recrystallization was not completed.
8. Ultrasonic test results correlated with those of earing tests in terms of both earing strength and earing position relative to the rolling direction.
9. R ratio results presented here agreed with those of Wilson and Bulter's satisfactorily.

V. 2. Projections

1. The tensile properties showed little variation in 0° , 45° , and 90° relative to the rolling direction (RD), which may imply that 22.5° , 67.5° to the RD should be investigated.
2. $\{200\}$ pole figures for the recrystallized state should be determined to understand the cube component contribution to the final cold rolled state.
3. Substructural variations in the different directions to the RD might be the real cause of anisotropy of earing problem. In other words, microbands, and shearbands resulting from the interactions between the particles, especially secondary particles, with defects like dislocations should be examined by TEM and other means. Recently, for example, Hjelen and Nes [60] used secondary electron channeling contrast imaging, and back scattered channeling imaging on TEM to get the cube grain locations after recrystallization of commercial Al alloys (1100 series). They also did $\{200\}$ pole figures within the recrystallized layers, then correlated the pole figure with the cube grain located by the special TEM techniques mentioned above.
4. Homogenization for 3104 needs to be investigated to find out the maximum cube texture formation temperature [18].
5. From a practical point of view, in order to increase cube texture after the cold rolling and intermediate annealing, so that the final H19 state can have the best combination of cube texture

and rolling texture, the following processing is suggested:

HR + BA {670, 650 °F} + CR88

HR + CR40 + BA{630, 650, 670 °F} + CR88

HR + CR45 + BA{630, 650, 670 °F} + CR88

HR + CR50 + BA{630, 650, 670 °F} + CR88

REFERENCES

- [1]. Metals Handbook, 9th Ed, Vol. 2:3-23 American Society for Metals, Metals Park, Ohio (1985).
- [2]. Metals Handbook, 8th Ed, Vol. 8:262 American Society for Metals, Metals Park, Ohio (1973).
- [3]. Metals Handbook, 8th Ed, Vol. 8:261 American Society for Metals, Metals Park, Ohio (1973).
- [4]. T. K. McGill-Taylor, Master's thesis, University of Tennessee, Knoxville (1989), Chapt. 2 and Chapt. 3, pp. 3-48.
- [5]. Metals Handbook, 8th Ed, Vol. 8:395 American Society for Metals, Metals Park, Ohio (1973).
- [6]. Metals Handbook, Desk Edition, D:6.31, American Society for Metals, Metals Park, Ohio (1985).
- [7]. E. J. Westerman and D. I. Smith, Effects of Preheating on 3104 Alloy Ingot Microstructure, Kaiser Aluminum and Chemical Corporation, West Virginia (1986).
- [8]. The Aluminum Association's Task Group on Segregation in Thick Sections, Macrosegregation in Wrought Aluminum Alloys, *Light Metal Age*, 44:5-9 (1986).
- [9]. R. V. Tilak, and J. G. Morris, Studies of the Effect of Thermomechanical Treatments on the Supersaturation Content of Strip-cast Aluminum alloy 3004, *Materials Science and Engineering*, 73:139-150 (1985).
- [10]. H. D. Merchant, J. G. Morris, and D. S. Hodgson, Characteristics of Intermetallics in Aluminum Alloy 3004, *Materials Characterization*, 25:339-373 (1990).

- [11]. H. D. Merchant, T. Z. Kattamis, and G. Scharf, Homogenization of Aluminum Alloys, in *Homogenization and Annealing of Aluminum and Copper Alloys*, (H. D. Merchant, J. Crane and E. H. Chia, eds.), The Metallurgical Society, Warrendale (1988), pp. 1-52.
- [12]. H. S. Goodrich, % Earing vs Preheat Cycle, *Research Report*, Ravenswood, West Virginia (1985).
- [13]. J. E. Hatch, *Aluminum, Properties and Physical Metallurgy*, American Society for Metals, Metals Park, Ohio (1984).
- [14]. A. Karlson, A. Oscarsson, B. Lehtinen, and W. B. Hutchinson, Influence of Homogenisation on Structure and Earing in Aluminium Alloy 3004, in *Homogenization and Annealing of Aluminum and Copper Alloys*, (H. D. Merchant, J. Crane and E. H. Chia, eds.), The Metallurgical Society, Warrendale (1988), pp. 99-116.
- [15]. L. Chen and J. G. Morris, Precipitation Behavior of Strip Cast 3004 Aluminum Alloy, *Src. Met.* 18:1365-1370 (1984).
- [16]. K. Fukada, M. Mizouchi and T. Kajiyama, Effect of Heating Rate and Temperature on Annealing Textures of 3004 Aluminum Alloy, in *Aluminum Alloys Their Physical and Mechanical Properties, Volume I*, University of Virginia, Charlottesville, Virginia (1986), pp. 483-497.
- [17]. P. M. B. Rodrigues, H. Bichsel and P. Furrer, Cube Texture Control in Aluminium Sheet and Foil Products, in *Textures in Non-Ferrous Metals and Alloys*, (H. D. Merchant and J. G. Morris, eds.), The Metallurgical Society, Warrendale (1985), pp. 45-59.
- [18]. Y. Kitao, E. Usai and T. Inaba, Effect of Homogenization

- Temperature and Annealing on the Annealed and Cold Rolled Texture in Alloy 3004, *Kobe Res. Dev.*, 32:2-27(1982).
- [19]. O. S. Es-Said and J. G. Morris, Deformation and Annealing Textures Produced in Strip Cast 3004 Aluminum Alloy, in *Aluminum Technology '86* (T. Sheppard, ed.), The Institute of Metals, London (1986), pp. 333-338.
- [20]. K. Ito, Effects of Fine Particles on Formation of Recrystallization Structures and Textures of Aluminum Alloys, in *Homogenization and Annealing of Aluminum and Copper Alloys* (H. D. Merchant, J. Crane and E. H. Chia, eds.), The Metallurgical Society, Warrendale (1988), pp. 169-179.
- [21]. R. D. Doherty, Nucleation, in *Recrystallization of Metallic Materials* (F. Haessner, ed.), Dr. Riederer Verlag GmbH, Stuttgart (1978), Chapt. 3, pp. 23-59.
- [22]. H. P. Stuewe, Driving and Dragging Forces in Recrystallization, in *Recrystallization of Metallic Materials* (F. Haessner, ed.), Dr. Riederer Verlag GmbH, Stuttgart (1978), Chapt. 2, pp. 11-21.
- [23]. E. Nes, The Effect of a Fine Particle Dispersion on Heterogeneous Recrystallization, *Acta Met.*, 24:391-398 (1976).
- [24]. E. Nes, Grain Size and Texture Control in Commercial Aluminium Alloys, in *Microstructural Control in Aluminum Alloys: Deformation, Recovery and Recrystallization* (E. H. Chia and H. J. McQueen, eds.), The Metallurgical Society, Warrendale (1986), pp. 95-108.
- [25]. E. Hornbogen and U. Koester, Recrystallization of Two-Phase Alloys, in *Recrystallization of Metallic Materials* (F. Haessner,

- ed.), Dr. Riederer Verlag GmbH, Stuttgart (1978), Chapt. 8, pp. 159-193.
- [26]. K. Luecke and O. Engler, Effects of Particles on Development of Microstructure and Texture during Rolling and Recrystallization in FCC Alloys, *Materials Science and Technology*, 6-11:1113-1130 (1990).
- [27]. I. L. Dillamore, Recrystallization Processing, in *Recrystallization of Metallic Materials* (F. Haessner, ed.), Dr. Riederer Verlag GmbH, Stuttgart (1978), Chapt. 10, pp. 223-249.
- [28]. H. J. McQueen, H. Chia, and E. A. Starke, The Microstructural Mechanisms in Dilute Al-Fe Conductor Alloys, in *Microstructural Control in Aluminum Alloys: Deformation, Recovery and Recrystallization* (E. H. Chia and H. J. McQueen, eds.), The Metallurgical Society, Warrendale (1986), pp. 1-18.
- [29]. F. J. Humphreys and D. J. Jensen, Structure and Texture Evolution during the Recrystallization of Particle Containing Materials, in *Annealing Processes - Recovery, Recrystallization and Grain Growth* (N. Hansen, D. J. Jensen, T. Leffers and B. Ralph, eds.), Risø National Laboratory, Roskilde, Denmark (1986), pp. 93-106.
- [30]. E. Hornbogen and H. Kreye, Influence of Second Phase Particles on Recrystallization, in *Textures in Research and Practice*, (J. Grewen and G. Wassermann, eds.), Springer-Verlag, Berlin (1969), pp. 274.
- [31]. G. E. Dieter, *Mechanical Metallurgy*, McGraw-Hill, London (1988), pp. 526-529.

- [32]. P. A. Hollinshead and T. Sheppard, Development of Rolling Textures in Al Alloy 3004 Subjected to Varying Hot-Rolling Deformation, *Metallurgical Transactions A*, 20A:1495-1507 (1989).
- [33]. W. B. Hutchinson, A. Oscarsson, and A. Karlsson, Control of Microstructure and Earing Behaviour in Aluminium Alloy AA3004 Hot Bands, *Materials Science and Technology*, 5:1118-1127 (1989).
- [34]. Y.-L. Liu, L. Delaey and J. P. Baekelandt, Substructure Development and Properties in Cold Rolled Al Alloy 3004, in *Al technology '86* (T. Sheppard, ed.), The Institute of Metals, London (1986), pp. 211-215.
- [35]. G. E. Dieter, *Mechanical Metallurgy*, McGraw-Hill, London (1988), pp. 671-678.
- [36]. R. L. Whiteley, How Crystallographic Texture Controls Drawability, *Metal Progress*, Nov.:81-84 (1968).
- [37]. D. V. Wilson and R. D. Butler, The Role of Cup-Drawing Tests in Measuring Drawability, *J. Inst. of Metals*, 90:473-483 (1962).
- [38]. I. L. Dillamore, and W. T. Roberts, Preferred Orientation in Wrought and Annealed Metals, *Metallurgical Reviews*, 10(39):271-380 (1965).
- [39]. C. S. Barrett, and T. B. Massalski, *Structure of Metals*, McGraw-Hill, New York (1966), Chapt. 9, pp. 193-221.
- [40]. R. J. Roe, Description of Crystallite Orientation in Polycrystalline Materials. III. General Solution to Pole Figure Inversion, *J. Appl. Phys.*, 36(6):2024-2031 (1965).

- [41]. K. H. G. Ashbee, and J. P. Sargent, Stereoscopic Presentation of Rodrigues Vector Representation of the Full Three-Dimensional Disorientation of Iron Crystals by Rolling, *Metallurgical Transactions A*, 21A:253-255 (1990).
- [42]. A.V. Clark Jr, R. C. Reno, R. B. Thompson, J. F. Smith, G. V. Blessing, R. J. Fields, R. P. Delsanto, and R. B. Mignona, Texturing Monitoring in Aluminum Alloys: A Comparison of Ultrasonic and Neutron Diffraction Measurement, *Ultrasonics*, 26:189-197 (1988).
- [43]. T. Sheppard, M. A. Zaidi, P. A. Hollinshead, and N. Raghunathan, Structural Evolution During the Rolling of Aluminium Alloys, in *Microstructural Control in Aluminum Alloys: Deformation, Recovery and Recrystallization* (E. H. Chia and H. J. McQueen, eds.), The Metallurgical Society, Warrendale (1986), pp. 19-43.
- [44]. H. Hu, Texture of Metals, *Texture*, 1:238-239 (1974).
- [45]. W. G. Fricke Jr, M. A. Prezystupa and F. Barlat, Modeling Mechanical Properties from Crystallographic Texture of Aluminium Alloys, in *Aluminum Technology '86* (T. Sheppard, ed.), The Institute of Metals, London (1986), pp. 310-316.
- [46]. T. C. Sun, J. G. Morris and H. D. Merchant, Effect of Mn Supersaturation on Texture and Earing Behavior of Aluminum Alloys, in *Textures in Non-Ferrous Metals and Alloys* (H. D. Merchant and J. G. Morris, eds.), The Metallurgical Society, Warrendale (1985), pp. 79-98.

- [47]. I. L. Dillamore and W. T. Roberts, Preferred Orientation in Wrought and Annealed Metals, *Metallurgical Reviews*, 10-39:271-379 (1965).
- [48]. I. L. Dillamore and W. T. Roberts, Rolling Textures in f.c.c. and b.c.c. Metals, *Acta Met.*, 12:281-293 (1964).
- [49]. I. L. Dillamore, Factors Affecting the Rolling Recrystallization Textures in f.c.c. Metals, *Acta Met.*, 12:1005-1014 (1964).
- [50]. T. Leffers, A Modified Cross-Slip Theory for the Rolling Texture of Face-Centered Cubic Metals and Alloys, in *Textures in Research and Practice*, (J. Grewen and G. Wassermann, eds.), Springer-Verlag, Berlin (1969), pp. 120-129.
- [51]. K. Luecke and J. Hirsch, Rolling and Recrystallization Textures of Aluminium, in *Al Technology '86* (T. Sheppard, ed.), The Institute of Metals, London (1986), pp. 267-279.
- [52]. C. S. Barrett, and T. B. Massalski, *Structure of Metals*, McGraw-Hill, New York (1966), Chapt. 20, pp. 541-567.
- [53]. R. Sundberg, The Occurrence of a "Brass-type" Texture in Aluminium, in *Textures in Research and Practice* (J. Grewen and G. Wassermann, eds.), Springer-Verlag, Berlin (1969), pp. 130-135.
- [54]. H. Hu, Nucleation of Recrystallization for Cube Texture Formation in f.c.c. Metals, in *Annealing Processes - Recovery, Recrystallization and Grain Growth* (N. Hansen, D. J. Jensen, T. Leffers and B. Ralph, eds.), Risø National Laboratory, Roskilde, Denmark (1986), pp. 75-92.
- [55]. G. F. Vander Voort, *Metallography Principles and Practice*,

McGraw-Hill, New York (1984), Append. I, pp. 610-711.

- [56]. K. R. Brown, Factors Affecting Texture Development in Rolled Aluminum Commercial Alloys, in *Textures in Non-Ferrous Metals and Alloys*, (H. D. Merchant and J. G. Morris, eds.), The Metallurgical Society, Warrendale (1985), pp. 131-143.
- [57]. B. D. Cullity, *Elements of X-Ray Diffraction*, Addition-Wesley Publishing Company, Inc., Philippines (1978), pp. 295-323.
- [58]. W. Precht, L. Christodoulou and F. Lockwood, A Method of Easy Assessment of Crystallographic Texture and Earing in 3004 Aluminum, in *Textures in Non-Ferrous Metals and Alloys*, (H. D. Merchant and J. G. Morris, eds.), The Metallurgical Society, Warrendale (1985), pp. 223-227.
- [59]. K. R. Brown, Heterogeneous Deformation and Texture Development in Rolled Aluminum Alloys, in *Textures in Non-Ferrous Metals and Alloys*, (H. D. Merchant and J. G. Morris, eds.), The Metallurgical Society, Warrendale (1985), pp. 117-129.
- [60]. J. Hjelen and E. Nes, Nucleation and Growth of the Cube Texture Component during Recrystallization of Aluminum, in *Annealing Processes - Recovery, Recrystallization and Grain Growth* (N. Hansen, D. J. Jensen, T. Leffers and B. Ralph, eds.), Risø National Laboratory, Roskilde, Denmark (1986), pp. 367-372.

APPENDICES

Appendix 1

Tensile test data

cond.	Tensile properties, yield and tensile strength, (YS, TS, Mpa/ksi), and Elongation (El. %)									
	YS-0°	YS-45°	YS-90°	TS-0°	TS-45°	TS-90°	El.-0°	El.-45°	El.-90°	
AR#1	151/ 21.9	148/ 21.4	158/ 22.9	201/ 29.1	191/ 27.7	201/ 29.1	21.0	21.0	18.0	
AR#2	152/ 22.0	143/ 20.7	155/ 22.5	201/ 29.2	190/ 27.6	198/ 28.8	22.0	20.0	18.0	
AR#3	150/ 21.7	145/ 21.0	152/ 22.0	202/ 29.3	190/ 27.6	200/ 29.0	21.0	20.0	18.0	
1-1#1	82/ 11.9	83/ 12.1	90/ 13.0	172/ 25.0	170/ 24.6	171/ 24.8	25.0	27.5	27.0	
1-1#2	88/ 12.7	83/ 12.1	91/ 13.2	172/ 25.0	170/ 24.6	170/ 24.6	25.0	28.0	27.0	
1-2#1	308/ 44.7	296/ 42.9	300/ 43.6	315/ 45.7	311/ 45.1	318/ 46.2	3.0	3.0	3.0	
1-2#2	308/ 44.7	295/ 42.8	298/ 43.2	316/ 45.9	309/ 44.8	320/ 46.4	3.0	3.0	3.5	
2-1#1	88.9/ 12.9	89.6/ 13.0	93.0/ 13.5	172.3/ 25.0	170.9/ 24.8	169.5/ 24.6	25.0	26.0	26.0	

2-1#2	89.6/ 13.0	87.5/ 12.7	90.9/ 13.2	173.6/ 25.2	170.2 /24.7	169.5/ 24.6	26.0	28.0	26.0
2-2#1	315.6 /45.8	300.4 /43.6	289.4/ 42.0	319.7/ 46.4	312.1 /45.3	310.1/ 45.0	2.0	2.0	2.5
2-2#2	316.9 /46.0	292.8/ 42.5	293.5/ 42.6	323.8/ 47.0	305.2/ 44.3	312.1/ 45.3	2.0	2.5	3.0
3-1#1	214.3 /31.1	215.7/ 31.3	213.6/ 31.0	226.7/ 32.9	227.4/ 33.0	232.9/ 33.8	7.0	7.0	8.0
3-1#2	216.3 /31.4	213.6/ 31.0	210.1/ 30.5	227.4 /33.0	228.8/ 33.2	229.4/ 33.3	7.5	7.0	8.0
3-2#1	77.2/ 11.2	79.2/ 11.5	76.5/ 11.1	173.6/ 25.2	167.4/ 24.3	166.7/ 24.2	24.0	24.0	23.0
3-2#2	76.5/ 11.1	78.6/ 11.4	77.2/ 11.2	172.3/ 25.0	166.1/ 24.1	167.4/ 24.3	25.0	24.0	24.0
3-3#1	290.8/ 42.2	272.8/ 39.6	281.8/ 40.9	299.0/ 43.4	286.6/ 41.6	295.6/ 42.9	3.0	3.0	3.0
3-3#2	293.5/ 42.6	277.0/ 40.2	286.0/ 41.5	301.8/ 43.8	290.1/ 42.1	301.8/ 43.8	3.0	3.0	3.0
4-1#1	230.8/ 33.5	230.8/ 33.5	225.3/ 32.7	240.5/ 34.9	240.5/ 34.9	238.4/ 34.6	8.0	9.0	8.0
4-1#2	230.8/ 33.1	223.2/ 32.4	228.7/ 33.2	240.5/ 34.9	236.3/ 34.3	239.8/ 34.8	7.0	9.0	8.0
4-2#1	82.0/ 11.9	83.4/ 12.1	82.7/ 12.0	172.3/ 25.0	170.2/ 24.7	172.9/ 25.1	24.0	25.0	23.0
4-2#2	81.3/ 11.8	83.4/ 12.1	83.4/ 12.1	174.3/ 25.3	171.6/ 24.9	173.6/ 25.2	24.0	25.0	26.0

Appendix 2

Earing test data

CODE	EARING (%)	DIRECTION
1-2#1	6.0	45°
2-2#1	5.0	45°
2-2#2	5.0	45°
3-1#1	12.0	45°
3-2#1	~	~
3-3#1	15.0	45°
4-1#1	12.0	45°
4-2#1	4.1	45°
4-2#2	5.3	45°
4-3#1	12.0	45°
5-2#1	2.0	90°
5-3#1	~	~
5-4#1	15.0	45°

Appendix 3

Programmings for Different Plane Diffractions on Rigaku diffractometer.

parameters	200 planes	220 planes	422 planes
mode	4	4	4
recycle	1	1	1
axis	1	1	1
start angle	43.8°	64.4°	136.3°
stop angle	45.3°	66.0°	138.0°
step width	4	4	4
full scale	6	6	6
preset time	6	6	6
chart speed	5	5	5
*2θ	44.76°	65.16°	137.8°

* θ is the Bragg angle of the diffracted plane {hkl}.

Appendix 4

R Values Measured from the Tensile Test Samples.

Condition	R Value	ΔR^*	$\bar{R}^{**}$
1-1 0°	0.19		
1-1 45°	0.20	0.02	0.21
1-1 90°	0.24		
1-2 0°	0.07		
1-2 45°	0.66	-0.49	0.42
1-2 90°	0.28		
2-1 0°	0.35		
2-1 45°	0.30	0.16	0.38
2-1 90°	0.56		

2-2 0°	3.02			
2-2 45°	2.46		-0.43	2.24
2-2 90°	1.03			
3-1 0°	0.39			
3-1 45°	0.26		0.11	0.31
3-1 90°	0.35			
3-2 0°	0.24			
3-2 45°	0.34		-0.16	0.26
3-2 90°	0.13			
3-3 0°	0.45			
3-3 45°	0.54		-0.19	0.45
3-3 90°	0.25			
4-1 0°	0.71			
4-1 45°	1.57		-0.78	1.18
4-1 90°	0.88			

4-2 0°	0.37			
4-2 45°	0.52	-0.19		0.43
4-2 90°	0.30			
4-3 0°	-5.01			
4-3 45°	7.67	-8.85		3.25
4-3 90°	2.66			
5-1 0°	0.19			
5-1 45°	0.20	0.02		0.21
5-1 90°	0.24			
5-2 0°	0.31			
5-2 45°	0.35	-0.03		0.34
5-2 90°	0.33			
5-3 0°	0.18			
5-3 45°	0.19	-0.05		0.17
5-3 90°	0.11			
5-4 0°	0.99			

5-4 45°	1.49	-0.65	1.16
5-4 90°	0.67		

* ΔR is the ratio of $(R_0 + R_{90} - 2 \cdot R_{45})/2$, so-called *planar anisotropy*.

** $\bar{R}$, or R_a , is the ratio of $(R_0 + R_{90} + 2 \cdot R_{45})/4$, so-called *normal anisotropy*.

VITA

Mingli Yu was born in Shandong Province, People's Republic of China (P.R.C.) on June 7, 1957. He graduated from the High School of Yingchun, Hulin County, Heilongjiang Province, P.R.C., in August of 1975, then worked as a plumber for 3 years. In 1978, he entered Fuxin Mining Institute (FMI), Liaoning Province, to pursue a degree in Engineering. In January of 1982, he received a Bachelor degree of Engineering from FMI, and immediately worked as a teaching assistant in the Teaching Group of Heat Treatment for Metals, Mechanical Engineering Department at FMI for 6 years. In August of 1988, he entered the Graduate School at the University of Tennessee, Knoxville, to study Metallurgical Engineering, and received his Master of Science degree in August, 1991.

简历

喻名立，男。1957年6月7日出生于山东曲阜，中华人民共和国。
1975年8月毕业于虎林县854农场中学。75年8月至78年1月，在854农场修建厂工作，管子工人。78年1月进入辽宁省阜新矿业学院机械工程系，学习机械工程。82年1月获学士学位，随即在阜新矿业学院任助教，热处理教研室，至88年秋天。届时奉调美国田纳西大学攻读冶金专业硕士学位。于91年8月获得硕士学位。