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THE EFFECTS OF ANNEALING ON THE MICROSTRUCTURE
AND MECHANICAL PROPERTIES OF 0.100-INCH-THICK
INGOT-SOURCE BERYLLIUM SHEET

MASTER

David L. Fergason

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ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

U. S. ATOMIC ENERGY COMMISSION
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David L. Fergason

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THE EFFECTS OF ANNEALING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF 0.100-INCH-THICK INGOT-SOURCE BERYLLIUM SHEET

David L. Ferguson

Abstract. The effects of various annealing cycles on the microstructure and room temperature mechanical properties of a typical as-rolled, 0.100-inch-thick, ingot-source beryllium sheet were investigated. Microstructures from heat-treated material having various yield strengths were quantitatively analyzed. The data indicate that a direct correlation exists between the percent of the large, unrecrystallized grains in the characteristically duplexed microstructures and the 0.2% yield strength.* It is suggested that the large grains have an orientation relationship to the plane of the sheet; this could explain their anisotropic deformation and recrystallization characteristics.

INTRODUCTION

Historically the development of beryllium has resulted in two kinds of commercially available material: ingot source and powder source. In the ingot source process relatively sound castings are enclosed in steel and then forged and/or rolled directly into sheet. In the powder source process the ingots are machined into chips, and ground into powder which is then vacuum-hot pressed into blocks.

The power wrought products are, in general, characterized by relatively high impurity content (1 to 5% BeO), strengths of ~55,000 psi minimum yield and ~70,000 psi ultimate, ductility of ~5%, a fine grain size (15 μm average) and a relatively high texturing tendency for a given reduction.¹ Ingot-sheet products, on the other hand, are characterized by higher purity levels (less than 0.50% total impurities), lower strengths (~30,000 psi yield and ~45,000 psi ultimate) higher ductility (~7%) and larger grain size (typically 40 μm). The lower impurity level and a lower texturing tendency are credited with the improved ductility in the thickness direction and the resulting improved formability of ingot versus powder metallurgy sheet. In view of the brittle history of beryllium it is not surprising that wrought ingot source products are used almost exclusively in the fully annealed condition to capitalize on ductility and formability. In contrast, powder products are rarely used in the fully annealed condition since optimum combinations of properties are achieved with partial anneals.

Annealing to achieve optimum formability with the ingot source product resulted in its being characterized by lower strength — a natural consequence of the fully annealed

condition. Therefore it has been less attractive from a strength-to-weight structural standpoint. Most beryllium products today are of powder origin.

In view of these considerations a major objective of this study was to investigate the effects of partial annealing treatments on the room temperature mechanical properties of ingot-source beryllium sheet. Specifically, this work was undertaken to determine what strength combinations could be achieved while maintaining a reasonable level of ductility.

Another major objective was to perform a quantitative microstructural analysis to determine whether a correlation between mechanical properties and microstructure could be observed for partially annealed wrought ingot beryllium sheet.

Before attempting to correlate mechanical properties with a microstructural phenomena it was first necessary to review the variables which control the mechanical properties of beryllium. These variables include the amount and kinds of impurities, the distribution of impurities, the grain size, the texture, the thermo-mechanical history, the amount of residual work, and the amount of surface damage from machining operations.

By limiting this study to a single beryllium sheet randomly selected from production material it was assured² that the starting material was uniform in chemistry (both in level, kinds, and distribution of impurities), microstructure, and texture; and that the material was representative of the production process. Partial annealing heat treatments could not be expected to change the level or kinds of impurities, and the surface damage effects could also be dismissed by following established industrial machining and chemical milling practices in the preparation of tensile bars.

The four remaining variables: grain size, texture, the amount of residual work, and impurity distribution, could be expected to change with heat treatment and affect the mechanical properties. The results of a literature survey follows:

Effect of Heat Treatment on Impurity Distribution

A number of investigators³⁻⁷ have observed a solutionizing-aging reaction between Fe and Al in beryllium. Carrabine et al.,⁴ have reported the influence of the Fe-Al interaction on the mechanical properties of powder source beryllium.

*The yield strength decreases linearly as the percentage of recrystallized microstructure increases.

Although powder source beryllium contains higher BeO content than ingot source material the remaining impurity levels, especially iron and aluminum, are similar. (Powder source scrap is used as feed for the ingot process.) Therefore a similar influence on ingot sheet properties can be expected.

Fraikor et al.,³ reviewed the literature on the Fe-Al precipitation reaction work and characterized the Dow Rocky Flats ingot source beryllium production sheet using electron microscopy and microprobe analyses. Their proposed precipitation reaction is summarized as follows:

*M = transition element such as Fe, Cr, Mn, or Ni.

Since the material used in this study was rolled to 780°C and air cooled to room temperature (a slow cool according to Fraikor⁸) the reaction predicts only AlMBe₄ precipitates to be present in the as-rolled condition. Since the approximate threshold temperature for excessive grain growth in ingot sheet is 800°C, all partial annealing treatments of this study were conducted at lower temperatures. The reaction predicts no change in the AlMBe₄ equilibrium for these heat treatments. Fraikor et al.,³ reported no change in impurity distribution after 20 hours at 780°C.

It was concluded that the partial annealing cycles used in this study did not change the impurity distributions. Therefore, no change in room temperature mechanical properties should be expected from this effect, since the

study was limited to a single production sheet. However, if samples of material had been used having different Fe/Al ratios, an effect on mechanical properties could have been expected.

Effect of Heat Treatment on Texture

Several investigators⁹⁻¹² have reported the texture behavior of ingot source beryllium sheet as influenced by heat treatment. In general, they found that the as-rolled texture of beryllium sheet was insensitive to heat treatment unless extensive grain growth occurred.

Since the times and temperatures selected for this study were not sufficient to cause extensive grain growth, it is assumed that the texture remained constant and did not bias the changes in mechanical properties caused by the partial anneals.

Effect of Heat Treatment on Recovery

G. L. Tuer et al.,¹³ reviewed the literature on recrystallization studies and presented x-ray line broadening evidence of recovery phenomena for powder and ingot source beryllium. Significant findings of their studies include the following:

- Recovery processes are essentially complete after relatively short times at temperatures that are considerably below the recrystallization temperature.
- The kinetic behavior of recovery is essentially independent of nucleation and growth processes. In beryllium the recovery process appeared to be complete before definite metallographic evidence of the recrystallization became apparent.
- Recovery and recrystallization processes have a close relationship to the modes of deformation which have operated in prior straining.

Of specific interest was their data which showed recovery processes to be approximately 95% complete after one hour at 650°C. The partial annealing cycles used in this study consisted of time and/or temperature combinations which would cause essentially complete recovery, except for samples tested in the as-rolled condition. In this study it was therefore concluded that the effects of recovery on the room temperature mechanical properties were essentially constant.

Effect of Grain Size: Recrystallization and Grain Growth

The influence of grain size on the mechanical properties of beryllium is well established for fully annealed material:

the metal follows a Hall-Petch-type relationship.^{14, 15} Since various strain states were intentionally induced in the following partial annealing characterization study, the material strain conditions were not constant and a Hall-Petch relationship was not expected.

The variation in grain size as the result of recrystallization and grain growth effects can be observed using light microscopy. Therefore optical microscopy was used to perform a quantitative analysis of the microstructure with confidence that the other variables of texture, impurity distribution, and recovery did not influence any correlation observed with mechanical properties.

HEAT TREATMENT AND MECHANICAL PROPERTY INVESTIGATION

Experimental Procedure

The 0.100-inch-thick, cross-rolled, ingot source material characterized in this study was randomly selected from the normal production stream with the exception that the material was not annealed after the last rolling pass. A summary of the fabrication history of the sheet selected is given in Table 1. A chemical analysis from samples of this material is listed in Table 2. The chemistry typical of the production material.

Tensile coupon locations were stenciled on the 0.102-inch by 17-inch by 52-inch sheet. The 1- by 4-inch coupons were removed from the sheet by band sawing and subsequently machined into tensile specimens using tracer-controlled side milling. Cuts of 0.015 inch were used in roughing, and finishing was done with successive passes of 0.010, 0.005, and 0.003 inch to minimize the amount and depth of surface damage in each specimen. All machining was done under cutting oil and is typical of Rocky Flats Division practice. The specifications for these flat tensile specimens are shown in Figure 1.

After machining, each specimen was chemically milled 0.003 to 0.004 inch per surface to remove the surface damage induced by the machining operation. The chemical milling also served as a quality control to detect latent surface defects which might have been caused by improper machining practices. Chemical milling was accomplished with a 20% HNO₃, 1% HF by volume, aqueous solution at room temperature.

Following the chemical milling, sets of three specimens were heat treated in a resistance-type vacuum tube furnace

Table 1. Thermal-Mechanical History of 0.100-Inch-Thick Ingot-Source Beryllium Sheet.*

Material: Vacuum-induction cast beryllium. Feed source is recycle ingot sheet scrap and/or S-200 type Powder scrap. Individual billet sizes are normally 9 inches square by 3.5 inches thick.

Can Material: Type 304 stainless steel (5/8-inch thick) assembled by electron beam welding and soaked 8 to 10 hours at 1040° ± 15°C.

A. Can Rolling Schedule:

Beryllium Thickness** Range (inch)	Reduction Per Pass (inch)	Temperature (°C ± 15°C)	Remarks
3.5 → 2.2	0.215	1040	Rotate 90° Between Passes
Reheat 25 minutes 2.2 → 1.14	0.200	980	Rotate 90° Between Passes
Reheat 25 minutes 1.14 → 0.81	0.150	870	Rotate 90° Between Passes
Reheat 15 minutes 0.81 → 0.55	0.130	870	Rotate 90° Between Passes
Reheat 15 minutes 0.55 → 0.35	0.100	870	Unidirectionally Roll
Reheat 15 minutes 0.35 → 0.250	0.065	780	Unidirectionally Roll
Reheat 20 hours		780	
Shear and Remove Stainless Steel		500- 700	See Note***
0.240 → 0.100	10%	780	Unidirectionally roll at 90° to last can rolling direction Reheats are 5-10 minutes between passes except for a one hour soak be- tween the fourth and fifth passes

*Taken from "Ingot Sheet Beryllium Fabrication" by J. L. Frankeny and D. R. Floyd, RFP-910, Rocky Flats Division, Dow Chemical U.S.A., February 9, 1968.

**Note that the dimensions refer to the beryllium thickness, not to the total can thickness.

***After the sheet has been annealed and decanned it is pickled in a 2% HF, 48% HNO₃, aqueous solution at room temperature to remove surface contaminants. Approximately 0.005 inch per surface is removed.

which had been preheated to temperature. The three specimens were positioned in the center of the three-inch-diameter by 10-inch-long heating zone and the furnace temperature was monitored with both the furnace control thermocouple and with a separate thermocouple attached to a potentiometer. The maximum time required to recover the preset furnace temperature after the samples had been placed was 5 minutes. This time includes an initial 30-second purge with argon just prior to evacuation. After each annealing cycle the furnace was back-filled with argon and the specimens were removed and air cooled to room temperature.

Table 2. Chemical Analysis of 0.100-Inch-Thick Ingot-Source Beryllium Sheet.

Impurity	Analysis (wt%)
Beryllium	99.37
Aluminum	0.073
Carbon*	.053
Calcium	.003
Cadmium	.001
Cobalt	.001
Chromium	.013
Copper	.008
Iron*	.151
Magnesium	.003
Manganese	.012
Molybdenum	.003
Oxygen**	.005
Nitrogen*	.007
Nickel	.025
Lead	.001
Silicon	.040
Titanium	.015
Tungsten	.020
Zinc	.010

*Chemical analysis; all others by spectroscopic analysis.

**Neutron activation analysis of 0.100-inch-thick ingot-source beryllium sheet.

Before the heat-treated tensile specimens were tested, each specimen was again chemically milled 0.003 to 0.004 inch per surface as a pickling treatment and as a precautionary measure to ensure that all residual surface damage effects had been removed.

The problems associated with the effects of machine damage have been reported by Beitscher¹⁶ for ingot-source beryllium and by Ward et al.,¹⁷ and Mathews et al.,¹⁸ for powder source beryllium. The above sample preparation technique is typical of industry practices with the exception that ordinarily the tensile specimens are machined from annealed material and only etched 0.004 to 0.005 inches per surface to remove machine damage. In this study all samples were double-etched a total of 0.008-inch minimum per surface as described above.

The heat-treated specimens were tensile tested on an Instron machine using a crosshead speed of 0.005 in./min. which corresponds to a strain rate of 0.005 in./in./min.

Results and Discussion

The room temperature tensile properties from the heat treatment study are listed in Appendix I and the mean values are shown in Table 3. The data include 95% confidence limits.

The heat treatment study was performed primarily on longitudinal tensile bars (tensile axis parallel to the last rolling direction); although a few transverse properties were

Legend

A — Gage length	1.125 ± 0.005 in.
M — One-half total length	2.000 ± 0.020 in.
C — Width of grip section	0.850 ± 0.005 in. (note increased width)
W — Width of gage section	0.200 ± 0.002 in.
R — Radius of fillet	0.5 in. nominal
H — Distance from center of gage length to center of hole	1.400 ± 0.005 in.
D — Diameter of holes	fit for 3/8-in. pin

Notes: The ends of the reduced section shall not differ in width by more than 0.002 inch. There shall be a gradual taper in width from the ends to the center of from 0.002 to 0.002 inch. It is imperative that there be no undercutting of the width at the fillet radii.

The specimen shall be symmetrical about its longitudinal axis to within 0.001 inch.

The surfaces of the reduced section shall be finished to at least 32 rms. The surfaces of the grip section shall be at least 63 root-mean-square. All obvious scratches or nicks that are in the reduced section and are transverse to the longitudinal axis shall be removed.

Figure 1. Pin-Grip Sheet-Type Tensile Specifications.

determined for comparison in the as-rolled and most fully annealed (760°C/24 hours) conditions. The data show that the 760°C/24 hours anneal caused nearly identical responses in the two directions and the transverse/longitudinal property ratios remained approximately constant: 0.2% yield strengths, 0.98; ultimate strengths, 0.9; and elongations, 0.67. Therefore the main heat treatment study was performed on longitudinal tensiles, and it was assumed that after partial annealing treatments the transverse properties would exhibit the same response as the longitudinal properties.

ULTIMATE TENSILE STRENGTHS

The longitudinal ultimate tensile strengths shown in Table 3, were fairly insensitive to the various time/temperature combinations. All mean ultimate tensile

Table 3. Tensile Properties of Partially Annealed 0.100-Inch-Thick Beryllium Sheet.

Heat Treatment (°C)	0.2% Yield Strength (ksi)	Ultimate Tensile Strength (ksi)	Elongation (%)
As-rolled T	50.3 ± 0.88	57.2 ± 4.0	1.1 ± 0.70
As-rolled L	51.1 ± 1.2	62.3 ± 1.4	2.0 ± 0.93
760/24 hr T	30.7 ± 3.0	51.5 ± 4.4	3.9 ± 1.3
760/24 hr L	31.2 ± 3.5	58.0 ± 3.5	5.5 ± 0.46
650/2 hr L	46.1 ± 3.6	62.0 ± 3.6	3.6 ± 2.7
4 hr L	45.8 ± 0.53	61.6 ± 0.76	3.5 ± 0.80
6 hr L	46.3 ± 0.30	59.1 ± 2.3	2.4 ± 0.76
675/2 hr L	43.1 ± 7.6	58.1 ± 9.0	3.2 ± 1.8
4 hr L	42.2 ± 8.4	58.9 ± 15.5	3.7 ± 1.9
700/2 hr L*	42.4 ± 0.63	59.8 ± 1.1	3.9 ± 0.75
4 hr L	41.4 ± 2.5	60.4 ± 5.1	4.3 ± 3.0
725/2 hr L	40.1 ± 0.80	60.7 ± 3.6	4.4 ± 2.2
4 hr L	40.9 ± 2.1	61.3 ± 3.5	4.7 ± 1.7
760/1 hr L	39.7 ± 2.3	59.6 ± 7.2	3.9 ± 1.7
2 hr L	36.0 ± 1.4	59.2 ± 6.2	4.9 ± 2.4
6 hr L	32.4 ± 0.63	58.4 ± 6.1	4.9 ± 2.7
24 hr L	31.2 ± 3.5	58.0 ± 3.5	5.5 ± 0.46
775/15 min L	37.1 ± 1.7	60.8 ± 1.3	5.4 ± 0.57
30 min L	34.9 ± 0.92	58.2 ± 4.2	5.4 ± 1.2
1 hr L	36.5 ± 3.3	59.2 ± 7.0	4.9 ± 2.2

The data represent averages of three tests except where indicated, and includes 95% confidence limits.

*Average of two specimens.

strengths remained between a high of 62.3 ksi (as rolled) and a low of 58.0 ksi (760°C/24 hr). No significant trends were observed in the 95% confidence limits.

0.2% YIELD STRENGTHS

The 0.2% yield strength data indicate that the higher heat-treatment temperatures were more effective in lowering the yield strength than longer times at lower temperatures. The 0.2% yield strength was lowered approximately 20 ksi; that is, from 51.1 ksi (as rolled) to 31.2 ksi (24 hours at 760°C). The 95% confidence limits did not indicate any significant trends between the lesser and more fully annealed tensiles; which indicates that no loss in reproducibility of results (or increase in data scatter) was observed in the less-annealed conditions.

ELONGATION AT FRACTURE

A comparison of the elongation data indicates an expected behavior: the elongations increase as the spread between the ultimate strength and the yield strength increases. With the ultimate strength remaining fairly constant throughout this study and the effects of increased annealing being shown by the lowering of the yield strength, the elongations increased from 1.1% (as rolled) to 5.5% (after 24 hours at 760°C). Again, no trends were seen in comparisons of the 95% confidence limits.

Five different yield strength conditions were selected from the mechanical property data for a statistical microstructural analysis. The selected yield strengths and corresponding heat treatments are listed in Table 4.

Table 4. Selected Yield Strengths for Microstructural Analysis.

Heat-Treatment Condition	0.2% Yield Strength (ksi)
As Rolled	51.1 ± 1.2
650°C/4 Hours	45.8 ± 0.53
700°C/2 Hours	42.4 ± 0.63
775°C/1 Hour	36.5 ± 3.3
760°C/6 Hours	32.4 ± 0.63

STATISTICAL CALCULATIONS OF 0.2% YIELD STRENGTH DATA

A statistical comparison of 0.2% yield strength from the five selected heat treatments using the students "t" test indicated a minimum confidence level of 99% that the differences in means were real. A comparison of variances also showed a 99% confidence level that the variability in the five sets of data was the same. The results of the students "t" tests and the "F" tests for variance are listed in Appendix II.

MICROSTRUCTURAL ANALYSIS

Experimental Procedure

Microstructural samples were prepared from one end of a tested tensile bar representative of each of the five selected heat-treatment conditions. The location of these metallographic samples is illustrated in Figure 2. A comparison

Figure 2. Sketch of Microstructural Sample Location.

between samples taken from tested and untested tensile bars was made to determine whether tensile testing disturbed the microstructure. No evidence of pinhole distortion or adjacent microstructural disturbance was observed.

The metallographic samples were machined from the tensile bar ends and mounted in a suitable mounting material. Rough grinding and final polishing procedures are discussed in detail in Reference 19, and summarized in Appendix III. Sample preparation required considerable skill to obtain quality photomicrographs which are suitable for statistical analyses. This was particularly true when preparing samples of the more fully worked structures containing sub-or-tilt boundaries which were difficult to distinguish from low-angle grain boundaries.

After the samples had been prepared, three or four representative areas were photographed at 100X from each of the five selected samples. Negatives of these areas were used to make 2X prints (200X total magnification) measuring approximately 8 by 10 inches. In this manner, relatively large areas of the samples could be examined at a magnification sufficient to resolve the finer structural details.

The determination of grain-size distribution was accomplished by measuring individual grains from each microstructure. Since the microstructures contained both non-uniform structures and nonequiaxed grains, an individual grain area measurement was used based upon the overall grain length and a visually estimated average grain width. Approximately 400 to 600 grains were measured from three or four representative areas of each microstructure.

The length and width data were converted to grain area data and, with the aid of a computer, to $\log_e$ (Grain Area) data. A sample of the data is listed in Appendix IV. A computer program was also used to calculate the arithmetic means and standard deviations, and average grain length-to-width ratio (L/W) for each area of each of the five material conditions. These data are listed in Appendix V.

Results and Discussion

GENERAL METALLOGRAPHIC OBSERVATIONS

Representative microstructures from the five selected heat treatments are shown in Figures 3, 4, 5, 6, and 7, in order of decreasing yield strengths.

Figure 3. As-Rolled Microstructure.

Figure 4. Partially Annealed Microstructure, 650° C/4 Hours.

Figure 5. Partially Annealed Microstructure, 700°C/2 Hours.

Figure 7. Partially Annealed Microstructure, 760°C/6 Hours.

Figure 6. Partially Annealed Microstructure, 775°C/1 Hour.

The following observations were apparent:

1. The as-rolled microstructure, Figure 3, reflects the influence of rolling above the recrystallization temperature. The microstructure consists primarily of large, very irregularly distorted, nonuniform grains measuring $\sim 50 \mu\text{m}$ by $\sim 350 \mu\text{m}$, interspersed with areas of small, uniform, equiaxed grains measuring $\sim 15 \mu\text{m}$. The small grains also show evidence of prior work.
2. Progressive annealing, Figures 4 through 7 caused increased recrystallization and grain growth.
3. Recrystallization was heterogeneous. New grains occurred in clusters which grew in size while much of the structure was unaffected by the particular anneal.
4. The larger nonuniform grains appeared to be relatively insensitive to the annealing treatments.
5. The orientation of the lineal striations or "bend planes" in the larger grains (within $\pm 20^\circ$ to the normal to the plane of the sheet) suggests an orientation dependence which may explain deformation and recrystallization behavior. Bend planes are produced by nonpure shear slip on the (0001) plane which causes bending or rotation about an

axis in the (0001) plane. Bend planes in beryllium have been identified both by x-ray evidence and surface markings as {1120}, secondary prism planes.²⁰

STATISTICAL ANALYSIS

Histograms were constructed using the grain area and $\log_e$ (Grain Area) data to determine which statistic, arithmetic mean or geometric mean, was more descriptive of the microstructure. The class intervals for each of the five, grain-area histograms were arbitrarily chosen as one-fourth the arithmetic standard deviation of that microstructure. Similarly, the class intervals for the $\log_e$ (Grain Area) data were chosen as one-third the respective geometric standard deviation.

The grain area frequency histograms are shown in Figures 8A through 8E. The data for these curves are listed in Appendix VI. These histograms show all grain distributions to be positively skewed which, in combination with the large differences in the arithmetic standard deviations (see Table 5) indicates that a low confidence could be expected using arithmetic means for inter-distributional comparisons.

The $\log_e$ (Grain Area) versus frequency histograms are shown in Figures 9A through 9E and the corresponding data are listed in Appendix VII. From the bell-shaped distributions it can be concluded that the distributions can ordinarily be compared using the geometric mean and standard deviations.²¹ The standard relationship between mean grain size and mechanical properties is the Hall-Petch relationship which is given by:

$$\sigma_e = (\sigma_0)_e + k l^{-1/2}$$

where l is the mean grain size and σ is some mechanical property at constant strain, ϵ . Investigators^{14, 15} have shown that a Hall-Petch relationship is followed by beryllium. However the material evaluation in this study was purposely annealed to have different strain conditions. It

is therefore not surprising that neither the arithmetic nor the geometric mean data from Table 5 exhibit a $l^{-1/2}$ linear relationship with 0.2% yield strength. With respect to decreasing yield strength, the geometric mean grain area first decreases and then increases with progressive annealing. While the arithmetic mean does not show the increase, it does level off. This effect can be explained as competing effects of recrystallization and grain growth. Both recrystallization and grain growth are thermally activated, and there is no reason to expect the activation energies to be the same. The first four material conditions reflect the predominant influence of recrystallization which caused the reduction in the arithmetic and geometric mean area. However, the overriding influence of grain growth is observed in the fifth material condition (760°C/6 hours), where an increase in the geometric mean grain area is observed. Although the arithmetic mean is not as sensitive to the grain growth, further annealing would cause it to increase also.

Since the arithmetic and geometric means were not suitable for correlating grain area with yield strength, $\log_e$ (Grain Area) cumulative frequency distributions, listed in Appendix VII, were plotted to investigate the influence of the heat treatments on the entire grain size distributions. From these curves, shown in Figure 10, it is apparent that the various anneals produced no simple trends. The median $\log_e$ (Grain Area) values (shown at 0.5 cumulative frequency) also did not show any strong correlation with yield strength. However, closer inspection of the curves revealed a qualitative correlation above 0.85 cumulative frequency: higher yield strength material exhibited larger $\log_e$ (Grain Area) values in this region, suggesting that the larger grains were strongly influencing the 0.2% yield strength.

A more quantitative description of the grain distributions was obtained upon plotting the grain area cumulative frequency distributions on log-probability paper. The data are listed in Appendix VI and plotted in Figure 11. Log-probability paper is designed to produce a linear plot from cumulative frequency data of a simple log-normal distribution. Analyses of data show the following:

Table 5. Summary of Statistical Analysis of Grain Measurements.

0.2% Yield Strength (ksi)	Mean Arith. Grain Area ($\text{mm}^2 \times 10^{-6}$)	Arithmetic Standard Deviation	Geometric Grain Area ($\text{mm}^2 \times 10^{-6}$)	Geometric Standard Deviation	Grain L/W Ratio	Number of Grains
51.1	2663	5292.9	683	6.91	4.08	484
45.8	2070	4778.6	523	5.58	3.45	390
42.4	1944	3710.2	497	6.13	3.16	360
36.5	1176	3014.4	333	3.48	2.87	638
32.4	1177	2319	504	3.72	1.75	495

Figure 8. Grain Area Histograms.

A. As-Rolled

B. After 650°C/4 Hours

C. After 700°C/2 Hours

D. After 775°C/1 Hour

E. After 760°C/6 Hours

Figure 9. Log_e (Grain Area) Histograms.

Figure 10. Cumulative Frequency Distribution of $\log_{10}$ (Grain Area) Data.

Figure 11. Cumulative Frequency Distributions of Grain Area Data.

1. The as-rolled microstructure grain area distribution, shown in Figure 11, is not a simple log-normal distribution. Apparently the mixed grain sizes produced by reheating and rolling above the recrystallization temperature do not cause a log-normal distribution in the as-rolled condition.
2. The annealed microstructure distributions in Figure 11 show two intersecting straight lines which indicate a duplexed log-normal distribution.
3. As annealing progresses, the area percentage of total structure occupied by the smaller grain distribution increases and the two distributions tend to form one. The intersection of the two straight lines in the annealed material distributions in Figure 11 indicates the percent of the structure occupied by each distribution.
4. A graph showing the relationship between the percent of the structure occupied by the small grain distribution versus 0.2% yield strength exhibits a linear relationship. See Figure 12. The as-rolled material did not exhibit a distinguishable distribution separation and therefore could not be plotted.

The increase in the percent of microstructure occupied by the small grain distribution because of increased annealing is caused by recrystallization and grain growth. The reduction in 0.2% yield strength is directly proportional to the increase in strain-free material. However, the measured effect is not just caused by the increasing amount of new strain-free material because a grain size parameter, not a recrystallized percentage parameter, was measured. In the as-rolled microstructure, Figure 3, a number of the fine grains show evidence of distortion and residual work. Such grains are also present in some of the partially annealed microstructures, see Figures 4 and 5. During the anneals large unrecrystallized grains from the large grain distribution decompose by recrystallization or are consumed by grain growth of recrystallized grains. This effect has been previously observed by Bunce and Evans⁹ and Cheer and Lowe.¹¹ Obviously the large grains will be reduced in size until the unrecrystallized portion will belong to the small grain distribution. It follows that the percentage of the microstructure occupied by the small grain distribution should not be interpreted as the absolute percentage of strain-free metal, but rather as being proportional to the percentage of strain-free metal.

A second microstructural quantity may be used to predict 0.2% yield strength in partially annealed ingot source

Figure 12. Relationship Between 0.2% Yield Strength and the Microstructural Percent Occupied by the Small-Grain Distribution.

beryllium sheet. The ratio of average grain length-to-width (L/W) was found to exhibit an approximately linear correlation with 0.2% yield strength (see Figure 13). The data are listed in Appendices IV and V and summarized in Table 5.

The data show that the average L/W ratio was reduced from 4:1 in the as-rolled condition to 1.75:1 in the most fully annealed condition. Since fully annealed material would exhibit an average L/W ratio of 1:1, corresponding to 100% equiaxed grains, it is apparent that the most fully annealed of the five conditions (760°C/6 hours) is not fully annealed.

In comparison with the relationship obtained in Figure 12 between the percentage of structure occupied by the small grain distribution and 0.2% yield strength, the average L/W versus 0.2% yield strength correlation shows slightly more deviation from a linear behavior, particularly in the more fully annealed materials. This is probably caused by the influence of the different annealing temperatures causing nonproportional responses in grain growth and recrystallization. However, an average L/W ratio would be much

easier and faster to measure, using the Heyn technique,²² than would individual grain-size measurements for a routine process.

INFERENCES FROM MICROSTRUCTURAL ANALYSIS

Rolling Behavior

There are several effects which may be responsible for the heterogeneous rolling recrystallization behavior of ingot source beryllium sheet. As previously discussed, rolling is conducted at temperatures above the recrystallization temperature so that one would expect some recrystallization during reheats between passes and perhaps spontaneously during rolling. The recrystallized grains would be "softer" and tend to selectively deform on subsequent passes yielding a mixed, distorted structure. A second contribution to the heterogeneous uniform/nonuniform structure is believed to result from the single crystal hardness anisotropy which a number of investigators,²³⁻²⁶ have reported. Single

Figure 13. Relationship Between 0.2% Yield Strength and Average Grain Length-to-Width Ratio.

crystal hardness is two to three times greater, parallel to the C-axis than perpendicular to it, at temperatures at least as high as 700°C. Certain grains having their C-axis approximately normal to the plane of the sheet would resist rolling deformation more than grains having other orientations. The distribution of bend planes which indicate the trace of the {1120} planes appears to be within $\pm 20^\circ$ of the normal to the plane of the sheet for the large unrecrystallized grains (see Figures 3 through 7). This suggests such an orientation relationship may be responsible for the deformation behavior of ingot source beryllium sheet or vice versa. Such observations have also been made by Evans and Bunce,²⁷ Cheer and Lowe¹¹ and others.

Annealing Behavior

The 0.100-inch-thick ingot-source beryllium sheet exhibited a heterogeneous recrystallization response to the partial anneals of this study. Heterogeneous recrystallization can be expected from heterogeneous deformation; however, effects recently observed by Hulsey,²⁸ and previously observed by Tuer et al.,¹³ indicate that recrystallization tendencies are also dependent upon the orientations of the crystals during deformation rather than just the amount of deformation. Hulsey's work with single crystals showed that, after rolling pre-oriented beryllium single crystals which were canned in stainless steel, a nonuniform recrystallization response was observed. After one hour at 700°C the recrystallization response ranged from less than one percent to over 50%. After one hour at 800°C the recrystallization response ranged from less than 10% to almost 100% depending on crystal orientation. Although the single crystals were rolled at 540°C it seems quite feasible that similar behavior could be expected from grains with polycrystalline beryllium sheet rolled at 780°C. Again, the distribution of bend plane directions suggests that an orientation-dependent relationship, particularly with the large grains which resist recrystallization, is responsible for the heterogeneous response to annealing.

CONCLUSIONS

Heat Treatment and Mechanical Property Investigation

- The ultimate tensile strength of 0.100-inch-thick, ingot-source beryllium sheet was relatively insensitive to the partial anneals of this investigation; varying from 62.3 ± 1.4 ksi in the as-rolled condition to 58.0 ± 3.5 ksi after 24 hours at 760°C in vacuum.
- The 0.2% yield strength varied from 58.0 ± 3.5 ksi in the as-rolled condition to 31.2 ± 3.5 ksi after 24 hours at 760°C.

- Elongation values showed an increase from $2.0 \pm 0.93\%$ in the as-rolled condition to $5.5 \pm 0.46\%$ after 24 hours at 760°C.
- Good combinations of ultimate tensile strength and 0.2% yield strength coupled with good ductility were observed after various partial annealing treatments. For example after 4 hours at 725°C the following properties were obtained: 61.3 ± 3.5 ksi ultimate tensile strength, 40.9 ± 2.1 ksi 0.2% yield strength, and $4.7 \pm 1.7\%$ elongation.

Microstructural Analysis

- The 0.100-inch-thick, ingot-source beryllium used in this investigation exhibits heterogeneous grain deformation during rolling and heterogeneous recrystallization response during annealing.
- The as-rolled grain area distribution is positively skewed; non-normal and non-log-normal. The microstructure consists of mixed areas of large, distorted, unrecrystallized grains measuring $\sim 50\mu\text{m}$ by $\sim 350\mu\text{m}$ interspersed with areas of small, uniform, equiaxed grain clusters containing grains averaging about $15\mu\text{m}$. The small equiaxed grains show evidence of work indicating recrystallization prior to the last rolling pass.
- The large unrecrystallized grains in the as-rolled microstructure are relatively insensitive to recrystallization.
- The distribution of bend plane directions in the unrecrystallized large grains (within approximately $\pm 20^\circ$ to the sheet normal) suggests that the large grains have an orientation relationship with the plane of the sheet; this may explain their rolling and recrystallization behavior.
- The annealed microstructure exhibits duplex log-normal distributions. The small-grain distribution is proportional to the volume of strain-free grains and the large-grain distribution is composed of distorted, unrecrystallized grains.
- Increased annealing causes recrystallization and grain growth which increases the percentage of the microstructure occupied by the small grains and causes the two distributions to merge toward one log-normal distribution.
- Both the percentage of microstructure occupied by the small-grain distribution and the average grain length-to-width ratio show a linear relationship with 0.2% yield strength, since they vary directly with the percentage of strain-free microstructure.

Inferences from Microstructural Analyses

The heterogeneous rolling and recrystallization behavior of polycrystalline ingot-source beryllium sheet are attributed to an orientation relationship of the large grains in the sheet.

RECOMMENDATIONS

The work on rolling recrystallization of beryllium single crystals, Hulsey,²⁸ should be extended from 540°C to at least ~780°C, which is the rolling temperature used in the production of commercial ingot-source beryllium. This work would be useful in optimizing the rolling schedules used for polycrystalline beryllium sheet.

A second interesting study is suggested by the bend plane distributions observed in this study. A two dimensional investigation should be made to confirm the relationship between the bend plane distribution and the basal plane distribution.

Hot stage metallography on as-rolled beryllium sheet might be used to explore optimum time/temperature combinations which could be used to minimize grain size and optimize the mechanical properties of production material.

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APPENDIX I Room Temperature Tensile Data from Partially-Annealed Beryllium

Condition	0.2% Yield Strength (ksi)	Ultimate Strength (ksi)	Elongation (%)	Remarks	Condition	0.2% Yield Strength (ksi)	Ultimate Strength (ksi)	Elongation (%)	Remarks
As-Rolled (L)	50.9 50.8 <u>51.5</u> 51.1	62.1 62.8 <u>61.9</u> 62.3	2.1 2.3 <u>1.7</u> 2.0		725°C/4 hr (L)	40.6 41.7 <u>40.4</u> 40.9	62.1 61.9 <u>60.0</u> 61.3	5.3 4.6 <u>4.2</u> 4.7	
Standard Deviation (s.d.)	0.60	0.70	0.46		Standard Deviation (s.d.)	1.0	1.8	0.85	
As-Rolled (T)	50.5 50.5 <u>50.0</u> 50.3	55.9 58.5 <u>57.3</u> 57.2	0.8 1.2 <u>1.2</u> 1.1		760°C/1 hr (L)	39.0 40.5 <u>39.6</u> 39.7	60.0 61.7 <u>57.0</u> 59.6	4.4 4.1 <u>3.3</u> 3.9	
s.d.	0.44	2.00	0.35		s.d.	1.2	3.6	0.85	
650°C/2 hr (L)	46.3 45.9 — 46.1	61.8 62.2 — 62.0	3.5 3.8 — 3.6	Broke in pin hole	760°C/2 hr (L)	35.7 36.5 <u>35.7</u> 36.0	58.5 61.5 <u>57.6</u> 59.2	4.6 5.8 <u>4.3</u> 4.9	
s.d.	1.80	1.80	1.85		s.d.	0.70	3.1	1.2	
650°C/4 hr (L)	45.6 45.9 <u>45.9</u> 45.8	61.9 61.4 <u>61.6</u> 61.6	3.8 3.4 <u>3.3</u> 3.5		760°C/6 hr (L)	32.6 32.2 <u>32.3</u> 32.4	59.4 56.1 <u>59.7</u> 58.4	5.0 4.0 <u>5.8</u> 4.9	
s.d.	0.26	0.38	0.40		s.d.	0.32	3.0	1.4	
650°C/6 hr (L)	46.2 46.3 <u>46.4</u> 46.3	58.9 58.4 <u>59.9</u> 59.1	2.4 2.2 <u>2.7</u> 2.4		760°C/24 hr (L)	31.4 32.3 <u>30.0</u> 31.2	58.2 59.1 <u>56.8</u> 58.0	5.3 5.6 <u>5.5</u> 5.5	
s.d.	0.15	1.6	0.38		s.d.	1.8	1.8	0.23	
675°C/2 hr (L)	44.6 40.2 <u>44.5</u> 43.1	61.1 55.2 <u>57.9</u> 58.1	3.6 3.5 <u>2.5</u> 3.2		775°C/15 min (L)	37.5 36.7 — 37.1	60.5 61.1 — 60.8	5.2 5.5 — 5.4	Broke in pin hole
s.d.	3.8	4.5	0.90		s.d.	0.85	0.65	0.28	
675°C/4 hr (L)	43.3 43.3 <u>39.1</u> 42.2	61.5 62.1 <u>53.0</u> 58.9	4.2 3.9 <u>3.0</u> 3.7		775°C/30 min (L)	34.8 34.7 <u>35.2</u> 34.9	56.6 58.9 <u>59.1</u> 58.2	5.4 5.0 <u>5.8</u> 5.4	
s.d.	4.2	7.8	0.95		s.d.	0.46	2.1	0.60	
700°C/2 hr (L)	42.8 41.9 <u>38.3*</u> 42.4	58.7 60.9 <u>53.4*</u> 59.9	3.0 4.5 <u>3.6*</u> 3.8	Insufficiently etched to remove machine damage	775°C/1 hr (L)	35.4 37.6 <u>36.5</u> 36.5	56.9 61.5 <u>59.1</u> 59.2	4.1 5.5 <u>5.2</u> 4.9	
s.d.	0.32	0.60	0.38		s.d.	1.6	3.5	1.1	
700°C/4 hr (L)	41.4 41.9 <u>40.3</u> 41.2	58.5 61.6 <u>61.1</u> 60.4	5.3 4.3 <u>5.3</u> 5.0		775°C/2 hr (L)	29.8* 30.6* <u>29.7*</u> 30.3*	52.2* 53.4* <u>52.0*</u> 52.5*	4.4* 5.3* <u>5.0*</u> 4.9*	Insufficiently etched to remove machine damage
s.d.	1.3	2.5	1.5		s.d.	0.75*	1.2*	0.70*	
725°C/2 hr (L)	40.1 39.8 <u>40.3</u> 40.1	59.5 61.9 <u>60.6</u> 60.7	3.7 5.0 <u>4.9</u> 4.4		775°C/4 hr (L)	34.2 29.0* <u>32.0*</u> 34.2	58.9 50.8* <u>54.8*</u> 58.9	4.8 5.1* <u>5.7*</u> 4.8	Insufficiently etched to remove machine damage
s.d.	0.40	1.8	1.1		s.d.	—	—	—	

APPENDIX II

Statistical Calculations of Significance for 0.2% Yield Strength Data

Material Condition	Number of Samples	Sample Mean, ksi	Sample Variance S_i^2
As-Rolled	3	51.1	0.143
650°C/4 hr	3	45.8	0.030
700°C/2 hr	2	42.4	0.124
775°C/1 hr	3	36.5	1.210
760°C/6 hr	3	32.4	0.0432

A. "t" Test Results of Significance Between Two Sample Means (99% Confidence Level).

$$t_c = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{(N_1 - 1)S_1^2 + (N_2 - 1)S_2^2}{N_1 + N_2 - 2} \left(\frac{1}{N_1} + \frac{1}{N_2} \right)}}$$

If $t_c > t_a$, difference between sample means is real.

As-Rolled versus 650°C/4 hr

$$t_c = 22.0$$

$$t_a = 2.132$$

Difference is real

650°C/4 hr versus 700°C/2 hr

$$t_c = 13.4$$

$$t_a = 2.353$$

Difference is real

700°C/2 hr versus 775°C/1 hr

$$t_c = 7.93$$

$$t_a = 2.353$$

Difference is real

775°C/1 hr versus 760°C/6 hr

$$t_c = 5.54$$

$$t_a = 2.132$$

Difference is real

B. "F" Test Results of Significance Between Variances (99% confidence level)

$$F_c = \frac{S_1^2}{S_2^2}, \text{ where } S_1^2 > S_2^2$$

If $F_c < F_a$, there is no significant difference between variances.

As Rolled versus 650°C/4 hr

$$F_c = 4.78$$

$$F_a = 99.01$$

No significant difference in variances

650°C/4 hr versus 700°C/2 hr

$$F_c = 4.13$$

$$F_a = 4999$$

No significant difference in variances

700°C/2 hr versus 775°C/1 hr

$$F_c = 9.78$$

$$F_a = 4999$$

No significant difference in variances

775°C/1 hr versus 760°C/6 hr

$$F_c = 20.8$$

$$F_a = 99.01$$

No significant difference in variances

APPENDIX III

Summary of Metallographic Grinding and Polishing Procedures (Reference 19)

After the metallographic samples have been mounted in a suitable mounting material, rough polishing is performed on successive 60, 180, 240, 320, 400, 600, and 600 soft [Buehler (Microcut) paper; Adolf I. Buehler, Inc., Evanston, Illinois] abrasive papers using moderate pressure and liberal amounts of water for lubrication. It is recommended that the papers be changed frequently to prevent smeared metal, and approximately $\frac{1}{16}$ inch of material should be removed to ensure elimination of machine and grinding damage. Between each grit size it is also recommended that the specimens be etched for 10 seconds in a 10 vol% sulfuric acid aqueous solution.

Final polishing is accomplished using automatic polishing units of the rotary or vibratory type. Successive polishing steps utilize 6μ , 3μ , 1μ , and $\frac{1}{4}\mu$ diamond abrasives with a water-soluble lubricant and respective polishing times of 15, 15, 5, and 3 minutes. Specimens are etched with a 2% hydrofluoric acid – ethyl alcohol solution for five seconds after each final polishing step to aid elimination of smeared metal. The last polishing step was conducted with a $0.05\text{-}\mu\text{m}$ gamma alumina using a 0.33% aqueous solution of chromic acid for lubricant. The samples were vibratory polished for 24 hours and examined by polarized light.

APPENDIX IV

Sample of Grain-Measurement Data

A. As Rolled Microstructure – Area 1

Measured at 200X		Converted Area 10^{-6} mm^2	log (Grain Area)	Measured at 200X		Converted Area 10^{-6} mm^2	log (Grain Area)
length, mm	average width, mm			length, mm	average width, mm		
60	11	16500	9.71112	3.5	1.5	131.25	4.87710
40	12	12000	9.39266	23.5	6	3525	8.16764
14	4	1400	7.24423	3	2	150	5.01064
29	5.5	3987.5	8.29092	2	2	100	4.60517
3	3	225	5.41610	31	2.5	1937.5	7.56398
1.5	1	37.5	3.62434	3	3	225	5.41610
1	1	25	3.21888	15	10	3750	8.22951
20	4	2000	7.60090	13	3	975	6.88294
3	3	225	5.41610	24	5.5	3300	8.10168
14	5.5	1925	7.56268	13	3	975	6.88244
3	1.5	112.5	4.72295	10	3	750	6.62007
3	1.5	112.5	4.72295	2.5	1	62.5	4.13517
22	6	3300	8.10168	7.5	2	375	5.92693
1.5	1	37.5	3.62434	31	5	3875	8.2623
3	2.5	187.5	5.23378	24	6	3600	8.18869
14	4.5	1575	7.36201	7.5	3	562.5	6.33239
0.5	0.5	6.25	1.83258	34	4	3400	8.13153
24	3.5	2100	7.64969	67	8	13400	9.50301
6	4	600	6.39693	38	4	3800	8.24276
19.5	6	2925	7.98105	2	2	100	4.60517
14.5	3	1087.5	6.99164	3	0.5	37.5	3.62434
32	4	3200	8.07091	27	8	5400	5.59415
3.5	0.5	43.75	3.77849	8.5	4	850	6.74524
4	0.5	50	3.91202	19	4	1900	7.54961
12	2	600	6.39693	11	3.5	967.5	6.86953
13.5	1.5	506.25	6.22703	4	2	200	5.29832
11.5	3	862.5	6.75984	1	1	25	3.21888
18.5	3	1387.5	7.23526	6	2	300	5.29832
8	5	1000	6.90776	0.5	0.5	6.25	1.83258
17	5	2125	7.66153	0.5	0.5	6.25	1.83258
3.5	3.5	306.25	5.72440	3	2	150	5.01064
1.5	1.5	56.25	4.02981	17	2.5	1062.5	6.96838

APPENDIX V

Statistics From Computer Analyses of Grain Area Data

I. AS-ROLLED MICROSTRUCTURE.

Area	$\bar{X}_A^1$	S_A^2	$\ln(X_G)^3$	$\ln(S_G)^2$	N	L/W
1	2261.93	4201.1	6.39656	1.88509	127	4.34
2	2513.32	2840.2	6.25047	2.05781	126	4.39
3	3824.64	7661.0	7.01732	1.54273	105	3.42
4	2251.89	4340.4	6.44284	1.85064	126	4.09
All Areas	2663.78	5292.9 ⁴	6.52591	1.93318 ⁴	484	4.08

II. 650°C/4 HR MICROSTRUCTURE.

1	1954.78	3267.0	6.32095	1.71954	140	3.60
2	1930.03	3595.8	6.13514	1.80778	126	3.52
3	2341.48	6870.4	6.31460	1.68176	124	3.21
All Areas	2069.73	4778.6 ⁴	6.25890	1.73681 ⁴	390	3.45

III. 700°C/2 HR MICROSTRUCTURE.

1	2504.58	4341.2	6.48294	1.81958	123	3.18
2	2119.89	4239.6	6.24368	1.87903	114	3.38
3	1235.22	2256.0	5.90475	1.73977	123	2.93
All Areas	1943.66	3710.2 ⁴	6.20963	1.81201 ⁴	360	3.16

IV. 775°C/1 HR MICROSTRUCTURE.

1	1387.89	3411.8	5.94748	1.56280	207	2.50
2	1002.44	2229.8	5.86626	1.39375	140	2.89
3	1443.83	4443.6	5.75168	1.75764	141	3.05
4	805.234	1959.5	5.54400	1.55203	150	3.18
All Areas	1175.76	3014.4 ⁴	5.80942	1.24756 ⁴	638	2.87

V. 760°C/6 HR MICROSTRUCTURE.

1	1042.01	1875.9	6.20790	1.18659	195	1.49
2	1516.83	2840.2	6.51498	1.30712	124	2.25
3	1086.27	2375.3	6.03136	1.44857	176	1.69
All Areas	1176.69	2319.1 ⁴	6.22205	1.31479 ⁴	495	1.75

NOTES:

$$(1) \quad \bar{X}_A = \sum_{i=1}^n X_i \quad \text{Arithmetic Mean}$$

$$(2) \quad S_i = \left[\frac{\sum_{i=1}^n X_i^2 - (\sum_{i=1}^n X_i)^2 / n}{n - 1} \right]^{1/2} \quad \text{Standard Deviation}$$

$$(3) \quad X_G = (X_1 \cdot X_2 \cdot X_3 \cdots X_n)^{1/n} \quad \text{Geometric Mean}$$

$$(4) \quad (S_i)_p = \left[\frac{\sum_{i=1}^n (n_i - 1) S_i^2}{\sum_{i=1}^n (n_i - 1)} \right]^{1/2} \quad \text{Pooled Standard Deviation}$$

Specifically:

S_A = Arithmetic Standard Deviation

S_G = Arithmetic Standard Deviation of $\ln(X_G)$

$(S_A)_p$ = Pooled Arithmetic Standard Deviation

$(S_G)_p$ = Pooled Arithmetic Standard Deviation of $\ln(X_G)$

L/W = Average Grain Length-To-Width Ratio.

APPENDIX VI

Grain Area Histogram and Cumulative Frequency Distribution Data

A. AS-ROLLED MICROSTRUCTURE.

Limit on Grain Area Class Interval* (μm) ²	Frequency f_i	Mean Grain Area per Class Interval, A_i (μm) ²	Tot. Grain Area per Class Interval $A_j = f_i \cdot A_i$ (μm) ²	Cumulative Frequency $F_h = A_j/A_j$
0 - 18	20	9	180	0.000121
1782	313	900	281700	.190
3546	74	2664	197136	.322
5310	26	4428	115128	.400
7074	13	6192	80496	.454
8838	7	7920	55692	.491
10602	11	9720	106920	.563
12366	3	11484	34450	.586
14130	9	13248	119232	.666
15894	1	15012	15012	.676
17658	1	16776	16776	.688
19422	4	18540	74160	.738
21186	2	20304	40608	.765
22950	2	22068	44136	.795
24714	1	23832	23832	.811
26478	2	25596	51192	.845
28242	2	27360	54720	.882
30006	2	29124	58238	.921
31770	0	30888	0	.921
33534	0	32652	0	.921
35298	0	34416	0	.921
37062	1	36180	36180	.945
38826	0	37944	0	.945
40590	1	39708	39708	.972
42354	1	41472	41472	1.000

B. 650°C/4 HR MICROSTRUCTURE.

0-1274	272	637	173264	0.203
2867	42	2071	86982	.305
4460	20	2664	73280	.391
6053	10	5257	52570	.452
7646	13	6850	89050	.557
9239	13	8443	109759	.686
10832	4	10036	40140	.733
12425	6	11629	69774	.814
14018	2	13222	26444	.845
15611	1	14815	14816	.863
17204	2	16408	32816	.901
18797	1	18001	18001	.922
20390	0	19549	0	.922
21983	0	21187	0	.922
23576	1	22780	22780	.949

B. 650°C/4 HR MICROSTRUCTURE. (continued)

Limit on Grain Area Class Interval* (μm) ²	Frequency f_i	Mean Grain Area per Class Interval, A_i (μm) ²	Tot. Grain Area per Class Interval $A_j = f_i \cdot A_i$ (μm) ²	Cumulative Frequency $F_h = A_j/A_j$
25169	0	24373	0	.949
26762	0	25966	0	.949
28355	0	27559	0	.949
29948	0	29152	0	.949
31541	0	30745	0	.949
33134	0	32338	0	.949
34727	0	33931	0	.949
36320	0	35524	0	.949
37913	0	37117	0	.949
39506	0	38710	0	.949
41099	0	40303	0	.949
42692	0	41896	0	.949
44285	1	43489	43489	1.000

C. 700°C/2 HR MICROSTRUCTURE.

0- 88	63	44	2772	0.0037
1325	187	707	132209	.180
2562	38	1944	73872	.278
3799	17	3181	54077	.350
5036	17	4418	75106	.450
6273	8	5655	45240	.510
7510	5	6892	34460	.556
8747	4	8129	32516	.600
9984	5	9366	46830	.662
11221	4	10603	42412	.718
12458	2	11840	23680	.750
13695	2	13077	26154	.785
14932	2	14314	28628	.823
16169	0	15551	0	.823
17406	1	16788	16788	.845
18643	2	18025	36050	.893
19880	0	19262	0	.893
21117	0	20499	0	.893
22354	0	21736	0	.893
23591	1	22973	22973	.924
24828	0	24210	0	.924
26065	1	25447	25447	.958
27302	0	26684	0	.958
28539	0	27921	0	.958
29776	0	29158	0	.958
31013	0	30395	0	.958
32250	1	31632	31632	1.000

D. 775°C/1 HR MICROSTRUCTURE.

0- 176	224	88	19712	0.0256
1176	302	676	204152	.291

D. 775°C/1 HR MICROSTRUCTURE. (continued)

Limit on Grain Area Class Interval* (μm) ²	Frequency f_i	Mean Grain Area per Class Interval, A_i (μm) ²	Tot. Grain Area per Class Interval $A_j = f_i \cdot A_i$ (μm) ²	Cumulative Frequency $F_h = A_j/A_j$
2176	52	1676	87152	.404
3176	21	2676	56196	.477
4176	13	3676	47788	.539
5176	8	4676	37408	.588
6176	4	5676	22704	.618
7176	4	6676	26704	.652
8176	2	7676	15352	.672
9176	1	8676	8676	.684
10176	4	9676	38704	.734
11176	2	10676	21352	.762
12176	1	11676	11676	.777
13176	2	12676	25352	.810
14176	0	13676	0	.810
15176	4	14676	58704	.886
16176	0	15676	0	.886
17176	0	16676	0	.886
18176	1	17676	17676	.909
19176	0	18676	0	.909
20176	1	19676	19676	.935
21176	0	20676	0	.935
22176	0	21676	0	.935
23176	1	22676	22676	.964
24176	0	23676	0	.964
25176	0	24676	0	.964
26176	0	25676	0	.964
27176	0	26676	0	.964
28176	1	27676	27676	1.000

E. 760°C/6 HR MICROSTRUCTURE.

0- 17	6	8.5	51	0.0000579
790	299	403.5	120646	.177
1563	101	1176.5	118826	.356
2336	32	1949.5	62384	.450
3109	29	2722.5	78952	.567
3882	10	3495.5	34955	.619
4655	6	4268.5	25611	.657
5428	3	5041.5	15124	.680
6201	2	5814.5	11629	.697
6974	1	6587.5	6588	.707
7747	5	7360.5	36802	.762
8520	1	8133.5	8134	.774
9293	1	8906.5	8906	.787
10066	2	9679.5	19359	.816
10839	1	10452.5	10452	.831
11612	1	11225.5	11226	.848
12385	0	11998.5	0	.848
13158	0	12771.5	0	.848

E. 760°C/6 HR MICROSTRUCTURE. (continued)

Limit on Grain Area Class Interval* (μm) ²	Frequency f_i	Mean Grain Area per Class Interval, A_i (μm) ²	Tot. Grain Area per Class Interval $A_j = f_i \cdot A_i$ (μm) ²	Cumulative Frequency $F_h = A_j/A_j$
13931	1	13544.5	13544	.868
14704	1	14317.5	14318	.889
15477	0	15090.5	0	.889
16250	0	15863.5	0	.889
17023	1	16636.5	16636	.914
17796	1	17409.5	17410	.940
18569	0	18182.5	0	.940
19342	0	18955.5	0	.940
20115	1	19728.5	19728	.970
20888	1	20501.5	20502	1.000

*Class Intervals are centered about the mean grain area where S/3 was arbitrarily selected as the Class Interval.

APPENDIX VII

Log_e (Grain Area) Histogram and Cumulative Frequency Distribution Data

A. AS-ROLLED MICROSTRUCTURE.

Limit on Log _e (Grain Area) Class Interval*	Frequency f_i	Mean Log _e (Grain Area) per Class Interval, A_i	Total Class Interval Area $A_j = f_i \cdot A_i$	Cumulative Frequency $F_j = A_j / \Sigma A_j$
1.63437				
2.09826	15	1.86632	27.995	0.00889
.56215	5	2.33020	11.651	.01269
3.02604	2	.79410	5.5882	.01436
.48994	12	3.25798	39.096	.02677
.95383	18	.72188	66.994	.04804
4.41772	10	4.18516	41.858	.06133
.88162	23	.64966	106.94	.09528
5.34551	37	5.11354	189.20	.1553
.80940	32	.57744	178.48	.2120
6.27329	53	6.04132	320.19	.3137
.73719	46	.50522	299.24	.4087
7.20108	45	.96910	313.61	.5082
.66497	54	7.43300	401.38	.6356
8.12886	41	.89688	323.77	.7384
.59276	31	8.36078	259.18	.8207
9.05665	17	.82466	150.02	.8683
.52054	22	9.28856	204.35	.9332
.98443	11	.75244	107.28	.9672
10.44833	8	10.21634	81.731	.9932
.91222	2	.68022	21.360	1.0000

B. 650°C/4 HR MICROSTRUCTURE.

1.69978			0.0000	0.00000
2.13398	5	1.91688	9.5844	.00393
.56818	1	2.35108	2.3511	.00490
3.00238	19	.78528	52.920	.02661
.43658	1	3.21948	3.2195	.02792
.87080	6	.65368	21.922	.03691
4.30500	17	4.08788	69.494	.06542
.73920	15	.52208	67.831	.09324
5.17340	41	.95628	203.21	.1766
.60760	43	5.39048	231.79	.2716
6.04180	38	.82468	221.34	.3624
.47600	38	6.25888	237.84	.4600
.91020	34	.69308	227.56	.5533
7.34440	32	7.12728	228.07	.6469
.77860	20	.56148	151.23	.7090
8.21281	19	.99568	151.92	.7712
.64701	17	8.42988	143.31	.8300
9.08121	22	.86408	195.01	.9100
.51542	16	9.29828	148.77	.9710

B. 650°C/4 HR MICROSTRUCTURE. (continued)

Limit on Log _e (Grain Area) Class Interval*	Frequency f_i	Mean Log _e (Grain Area) per Class Interval, A_i	Total Class Interval Area $A_j = f_i \cdot A_i$	Cumulative Frequency $F_i = A_j / \Sigma A_j$
.94962	4	.73248	38.930	.9870
10.38382	1	10.16668	10.167	.9911
.81802	1	.60088	10.601	.9955
11.25223	1	11.03508	11.035	1.0000

C. 700°C/2 HR MICROSTRUCTURE.

1.45310				0.00000
.90611	5	1.67960	8.3980	.00378
2.35911	0	2.13260	0.0000	.00378
.81211	3	.58560	7.7568	.00726
3.26511	21	3.03860	63.811	.03596
.71812	10	.49160	34.916	.05166
4.17112	17	.94460	67.058	.08181
.62412	18	4.39760	79.157	.1174
5.07712	26	.85060	126.12	.1741
.53013	23	5.30360	121.98	.2290
.98313	32	.75660	184.21	.31181
6.43613	39	6.20960	242.17	.4207
.88914	36	.66260	239.85	.5286
7.34214	29	7.11560	206.35	.6214
.79514	26	.56860	196.78	.7098
8.24814	19	8.02160	152.41	.7784
.70114	21	.47460	177.97	.8584
9.15415	16	.92760	142.84	.9226
.60715	12	9.38060	112.57	.9733
10.06015	5	.83360	49.168	.9954
.51316	1	10.28660	10.287	1.0000

D. 775°C/1 HR MICROSTRUCTURE.

1.28936				
.68084	1	1.48510	1.4851	0.000519
2.07234	6	.87659	11.260	.00446
.46383	0	2.26809	0.0000	.00446
.85532	6	.65958	15.957	.01004
3.24681	25	3.05106	76.277	.03671
.63831	5	.44256	17.213	.04273
4.02980	19	.83404	72.847	.06820
.42129	14	4.22554	59.157	.08888
.81273	35	.61702	161.60	.1454
5.20428	46	.00852	230.39	.2260
.59577	53	5.40000	286.20	.3260
.98726	54	.79150	312.74	.4354
6.37880	55	6.18298	340.06	.5543
.77025	38	.57448	249.83	.6416
7.16174	37	.96596	257.74	.7318
.55323	30	7.35746	220.72	.8089

D. 775°C/1 HR MICROSTRUCTURE: (continued)

Limit on Log _e (Grain Area) Class Interval*	Frequency f_i	Mean Log _e (Grain Area) per Class Interval, A_i	Total Class Interval Area $A_j = f_i \cdot A_i$	Cumulative Frequency $F_i = A_j / \Sigma A_j$
.94472	19	.74894	147.23	.8604
8.33622	16	8.14044	130.25	.9060
.72771	6	.53192	51.192	.9239
9.11920	6	.92341	53.540	.9426
.51070	8	9.31490	74.519	.9686
.90219	5	.70640	48.532	.9856
10.29368	3	10.09792	30.294	.9962
.68517	0	.48938	0.0000	.9962
11.07667	1	.88087	10.880	1.0000

E. 760°C/6 HR MICROSTRUCTURE.

1.78463				
2.11333	2	1.94898	3.8980	0.00127
2.44202	0	2.27768	0.000	.00127
.77073	4	.60638	10.426	.00466
3.09942	0	.93508	0.000	.00466
.42812	8	3.26378	26.11	.01315
.75682	5	.59248	17.96	.01900
4.08552	13	.92118	50.98	.03558
.41421	7	4.24988	29.75	.04526
.74291	26	.57858	119.04	.08398
5.07160	27	.90728	132.50	.1271
.40031	24	5.23598	125.66	.1680
.72900	68	.56468	378.40	.2911
6.05770	35	.89338	206.27	.3582
.38640	41	6.22208	255.10	.4412
.71509	51	.55078	334.09	.5498
7.04379	67	.87948	460.92	.6998
.37249	32	7.20818	230.66	.7748
.70119	25	.53688	188.42	.8361
8.02989	22	.86558	173.04	.8924
.35858	14	8.19428	114.72	.9297
.68728	7	.52298	59.661	.9491
9.01598	7	.85168	61.962	.9693
.34468	5	9.18038	45.902	.9842
.67337	2	.50908	19.018	.9904
10.00207	3	.83778	29.513	1.0000

*Class intervals are centered about the mean log_e (Grain Area) where $S_G/4$ was arbitrarily selected as the class interval.