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FABRICATING VANADIUM SHEET TO IMPROVE SYMMETRY OF DRAWN PARTS

C. M. Edstrom

SUBJECT DESCRIPTORS

Drawing Vanadium
Earing
Forming Vanadium
Planer Isotropic Vanadium
Vanadium
Vanadium Earing
Vanadium Grainsize

ROCKWELL INTERNATIONAL
ENERGY SYSTEMS GROUP
ROCKY FLATS PLANT
P.O. BOX 464
GOLDEN, COLORADO 80401

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FABRICATING VANADIUM SHEET TO IMPROVE SYMMETRY OF DRAWN PARTS

C. M. Edstrom

ABSTRACT

The degree of anisotropy in vanadium sheet determines the amount of earing in formed parts and relates to the roundness of the formed part. Experiments with annealing and rolling practices to produce vanadium sheet were undertaken to reduce the earing tendencies of the metal. Earing in formed vanadium hemishells was reduced by cross rolling; and for unidirectionally rolled sheet, increased reduction per rolling pass and proper selection of intermediate annealing temperature reduced the earing. These modifications in the production steps did not adversely affect the grain size or formability of the vanadium, since these characteristics are mostly influenced by the final anneal.

OBJECTIVE

To minimize machining problems, it is desirable to produce formed vanadium parts with maximum roundness, i.e., minimum TIR (total indicated runout). The fabricating procedure for making the sheet should influence roundness of formed parts. However, any fabricating change should not adversely affect the grain size or formability (percent draw). Grain size should remain at ASTM 6 or finer, and the percent draw should remain at 48% or better, where

Percent draw is

$$(1.00 - \frac{\text{Punch Dia.} + \text{thickness}}{\text{Blank Dia.}}) \times 100\% = 48\%$$

INTRODUCTION

Roundness of drawn parts can be improved by achieving planer isotropic conditions in the starting sheet, so that strain ratios in the rolling direction, 90° to the rolling direction, and 45° to

the rolling direction are all equal to unity.¹

When the strains are not balanced, parts formed show four ears or lobes. If earing can be reduced, formed parts are more round, improving subsequent machining operations. A study was performed to determine if cross rolling vanadium would equalize the strain and thus reduce the earing experienced with unidirectionally rolled plate. The plate is turned 90° between each rolling pass during cross rolling.

Industry also has demonstrated with aluminum that placement of anneals, variation in anneal temperature, and rolling reductions between anneals can reduce earing for unidirectionally rolled sheet.^{2,3} Fabricating techniques influence the amount of earing since earing relates to texture. With primary rolling texture, the ears occur in the two planes 45° to the rolling direction. With annealing texture, the ears are in the planes 0° and 90° to the rolling direction. When the annealing and rolling textures are balanced, the tendency is to form eight ears, reducing the prominence of the ears.² With this information, the study was expanded to include rolling in one direction with variation in anneal temperatures before and after rolling and variations in reduction per rolling pass.

PROCEDURE

Vanadium was purchased in the as-rolled condition at 1-inch thickness and rolled at Rocky Flats to 0.125-inch thickness with fabrication variables shown in Table 1.

The vanadium used in this test was 99.85% pure as shown by the ingot chemistry reported in Table 2. It is desirable to experiment with high-purity vanadium because the higher the purity, the more difficult it becomes to produce fine grain size.⁴ If the fabrication procedure developed on this vanadium still produces fine grain size, then

standard chemistry vanadium should also produce fine grains.

Two 7- X 7 1/2- X 1-inch plates were cut, as shown in Figure 1, to provide ten blanks approximately 2 1/4 inches square. The fabrication tree for these sections is shown in Figure 2. The actual rolling schedules followed are presented in Table 3.

The rolled sheet was cut into 2-inch-diameter blanks, and these blanks were annealed prior to forming. Sketches of the punch and draw die used in the forming are shown in Figures 3 and 4, respectively. The punch diameter is 0.900 inch, and the blank thickness is 0.125 inch. Thus, the percent draw is:

$$\begin{aligned} & (1.00 - \frac{\text{Punch Dia.} + \text{Thickness}}{\text{Blank Dia.}}) \times 100\% \\ & = (1.00 - \frac{1.025}{2}) \times 100\% = 49\% \end{aligned}$$

After forming, peaks and valleys of the ears were measured to determine percent earing. Samples representing each blank drawn (thus, each fabricating condition) were also examined for grain size.

GRAIN SIZE RESULTS

The grain structure of the as-rolled 0.125-inch-thick vanadium provided some interesting observations. See Figure 5. The cross-rolled vanadium grain structure (Sections A, D, and H) shows both columnar grains and some grains of near-equiaxed size. The equiaxed grains within the cross-rolled structure substantiate the randomness of cross-rolling deformation since these grains are actually columnar with the major axis 90° to the plane of the sample. The columnar grains are also waved more in the cross rolled than in the unidirectionally rolled vanadium. (Compare Sections F and H.) Also, with increased reductions per pass, i.e., increased strain rates, the columnar grains show more wave. (Compare Sections A and D.) The effect of the anneal temperature before cold rolling is apparent in the as-rolled structure. Section B was annealed at 750 °C before rolling and the

columnar grains are very elongated, whereas Section G (annealed at 950 °C before rolling) has thicker columnar grains.

The grain sizes of the 0.125-inch-thick vanadium after the various final anneals are tabulated in Table 4. Transferring that data into graph form for the material unidirectionally rolled with 12 passes, Figure 6 shows that the final anneal has the greatest influence on final grain size. The lowest temperature final anneal (825 °C) did not fully recrystallize the vanadium, and the material annealed at the highest temperature (940 °C) had the largest grains. The initial anneal at the 1-inch plate thickness showed influence only at the high temperature. The final grain size of the product, which started with a two-hour initial anneal at 950 °C, is larger in Figure 6 than the other samples. However, the lowest initial anneal temperature (750 °C) did not provide the smallest final grain size.

Statistical Analysis 4 in Appendix A reports that the final anneal temperature has a statistically significant effect on grain size (at the alpha = 0.10 level). This confidence level qualifies that grain size has a 10% chance of not being affected by the final anneal temperature. Analysis 1, where influence of the initial anneal is considered along with the final anneal, does not provide enough statistical power to find a correlation between grain size and final anneal temperature.

Figure 7 shows that the rolling method may influence the grain size. During unidirectional rolling, the light reduction schedule (16 passes) produces grain sizes larger than those produced with heavier rolling schedules (12 passes and 8 passes). However, at the higher final anneal temperatures (920 and 940 °C), the most severe rolling schedule (8 passes) gave more grain growth than the least severe rolling schedule (16 passes). Figure 7 also indicates that the rolling procedure had greater influence on final grain size when the initial anneal temperature was 920 °C rather than 885 °C. None of these observations are supported statistically; however, a different display of the data (found in Table 3b of Appendix A) suggests that the interaction between rolling direction and number of passes may be important for grain size control.

TABLE 2. Chemistry of Vanadium

	Element	Ingot Analysis Results (wt %)			
		Specification Maximum	Top	Middle	Bottom
Cross rolled versus uniaxially rolled	H	0.005	<0.0005	<0.0005	<0.0005
	C	0.02	0.005	0.006	0.009
Four rolling reduction schedules	N	0.02	0.004	0.005	0.008
	O	0.025	0.017	0.017	0.021
• 22 passes, 7% average reduction per pass	Al	0.05	0.03	0.02	0.02
	Si	0.20	0.01	0.01	0.01
• 16 passes, 12% average reduction per pass	Fe	---	0.005	<0.005	<0.005
	Ni	---	<0.002	<0.002	<0.002
• 12 passes, 16% average reduction per pass	Cr	---	<0.002	<0.002	<0.002
	Fe + Ni + Cr	0.05	0.009	<0.009	<0.009
• 8 passes, 22% average reduction per pass	Nb	---	<0.02	<0.02	<0.02
	Mo	---	0.01	0.01	0.01
Variation in starting anneals	Ta	---	<0.02	<0.02	<0.02
	Nb + Mo + Ta	0.10	0.06	0.06	0.06
• 2 hr at 950 and 920 °C	Ti	---	<0.005	<0.005	<0.005
	Zr	---	<0.01	<0.01	0.02
• 1 1/2 hr at 885, 825, and 750 °C	Hf	---	<0.01	<0.01	<0.01
	Ti + Zr + Hf	0.050	<0.025	<0.025	0.033
Variation in final anneals	P	---	<0.003	<0.003	<0.003
	S	---	0.002	0.002	0.002
• 1 1/2 hr at 940, 920, 900, 885, and 825 °C	P + S	0.015	0.005	0.005	0.005
	B	---	0.0002	0.0002	0.0005
• 45 min at 885 °C	U	---	<0.0002	<0.0002	<0.0002
	V	99.6 min	>99.6	>99.6	>99.6

TABLE 3. Actual Rolling Schedules

Pass	22 Pass		12 Pass		8 Pass		16 Pass	
	Thickness (inch)	Reduction (%)	Thickness (inch)	Reduction (%)	Thickness (inch)	Reduction (%)	Thickness (inch)	Reduction (%)
1	0.940	= 6	0.875	= 12.5	0.850	= 15	0.900	= 10
2	0.895	= 5.8	0.750	= 14.3	0.700	= 17.7	0.810	= 10
3	0.865	= 3.4	0.650	= 13.7	0.575	= 17.9	0.730	= 10
4	0.820	= 5.5	0.550	= 15.4	0.450	= 21.8	0.660	= 10
5	0.770	= 6.1	0.475	= 13.7	0.350	= 22.3	0.590	= 10
6	0.685	= 4.8	0.400	= 15.8	0.250	= 28.6	0.530	= 10
7	0.645	= 6.3	0.350	= 12.5	0.175	= 30	0.470	= 10
8	0.600	= 7.2	0.300	= 14.3	0.125	= 28.6	0.410	= 10
9	0.555	= 7.2	0.250	= 16.7			0.360	= 12
10	0.515	= 7.0	0.200	= 20			0.310	= 14
11	0.475	= 8.0	0.160	= 20			0.270	= 13
12	0.430	= 9.1	0.125	= 22			0.230	= 15
13	0.390	= 9.3					0.200	= 13
14	0.360	= 8.7					0.170	= 15
15	0.335	= 6.2					0.145	= 17
16	0.295	= 12.2					0.125	= 14
17	0.255	= 13.6						
18	0.225	= 11.8						
19	0.190	= 16.0						
20	0.170	= 10.1						
21	0.135	= 20.6						
22	0.125	= 7.5						

TABLE 4. Grain Size Related to Final Anneal

Final Anneal (Time)	(°C)	Cross Roll (CR) or Unidirectional (U) Rolling Schedule (Passes)	(Type)	Initial Anneal (°C)	Sample Code*	Grain ASTM Size ^a
45 min.	885	22	CR	885	4A	7.5 ^b
45 min.	885	12	U	885	4E	8 ^b
45 min.	885	12	U	825	4C	7.5-8
45 min.	885	12	U	750	4B	6.5-8
45 min.	885	8	CR	885	4D	7.5 ^b
1 ½ hr.	825	22	CR	885	3A	8 ^b (90% recrys.)
1 ½ hr.	825	12	U	885	3E	8-8.5 ^b
1 ½ hr.	825	12	U	825	3C	7-8.5 ^b (99% recrys.)
1 ½ hr.	825	12	U	750	3B	8.5 ^b (95% recrys.)
1 ½ hr.	825	8	CR	885	3D	7.5-8
1 ½ hr.	885	22	CR	885	1A	7-7.5
1 ½ hr.	885	16	U	920	1I	4.5
1 ½ hr.	885	12	U	885	1E	7.5
1 ½ hr.	885	12	U	825	1C	5.5-7.5
1 ½ hr.	885	12	U	750	1B	7.5
1 ½ hr.	885	12	U	950	1G	6
1 ½ hr.	885	12	U	920	1F	6.5-7.5
1 ½ hr.	885	12	CR	920	1H	6.5-7.5
1 ½ hr.	885	8	U	920	1J	5
1 ½ hr.	885	8	CR	885	1D	7-7.5
1 ½ hr.	900	16	U	920	2I	4.5
1 ½ hr.	900	12	U	920	2F	6-7.5
1 ½ hr.	900	12	U	950	2G	6.5
1 ½ hr.	900	12	CR	920	2H	6.5
1 ½ hr.	900	8	U	920	2J	5.5-6.5
1 ½ hr.	920	22	CR	885	2A	6.5
1 ½ hr.	920	16	U	920	3I	4.5
1 ½ hr.	920	12	U	885	2E	7
1 ½ hr.	920	12	U	825	2C	6.5
1 ½ hr.	920	12	U	750	2B	6.5-7
1 ½ hr.	920	12	U	950	3G	5.5-6.5
1 ½ hr.	920	12	U	920	3F	6
1 ½ hr.	920	12	CR	920	3H	4-6.5
1 ½ hr.	920	8	U	920	3J	3.5
1 ½ hr.	920	8	CR	885	2D	6.5-7
1 ½ hr.	940	16	U	920	4I	4-6
1 ½ hr.	940	12	U	950	4G	4.5
1 ½ hr.	940	12	U	920	4F	5.5
1 ½ hr.	940	12	CR	920	4H	3.5
1 ½ hr.	940	8	U	920	4J	3

*See Figure 2.

^aFully recrystallized unless otherwise indicated. The low-temperature final anneals did not fully recrystallize the vanadium and, in many cases, did not fully soften the metal; therefore, the blank broke during forming.

^bBlank broke during forming, indicating that the temperature was inadequate for a full anneal.

Figure 8 provides a representative range of grain sizes developed in the experiment. Use this figure to visualize the grain size recorded in Table 4 for various samples.

GRAIN SIZE DISCUSSION

Vanadium grain size results substantiate that grain size is determined not only by the time and temperature of the anneal, but also the amount of prior cold work. Therefore, grain size is determined by the summation of strain energy from rolling and thermal energy of the anneal. When the strain energy in the metal is high, recrystallization occurs at a lower annealing temperature. As this experiment showed, the strain energy is influenced by the total work in the vanadium and the strain rate of that work. Since the total percentage of rolling reduction was kept constant throughout the experiment, the total work varied only because of the initial anneal.

At the low temperature, initial anneal (750 °C) residual strain remained in the vanadium; and at the high temperature (950 °C) anneal, no strain remained. When residual strain was added to the strain of rolling, the total work increased; and this lowered the temperature required to obtain recrystallization during the final anneal. When the required recrystallization temperature was exceeded, grain growth occurred.

The rate of rolling reduction or strain rate also affected the strain energy in the vanadium. With higher rolling strain rates (i.e., less rolling passes to reach final thickness), the recrystallization temperature lowered. When that temperature was exceeded, grain growth occurred.

EARING RESULTS

The forming and earing results are shown in Table 5. The comparison of a formed part with earing and one with minimum earing is shown in Figure 9. The percent earing is determined by:

$$\frac{\text{Average height of ear} - \text{average height of valley}}{\text{Average height of valley}} \times 100 = \text{earring}$$

TABLE 5. Earing Related to Rolling Schedule

Cross Roll (CR) or Unidirectional (U) Rolling Schedule		Initial Anneal	Final Anneal		Percent Earing	Sample Code*
(Passes)	(Type)	(°C)	(Time)	(°C)		
8	CR	885	1 ½ hr.	885	1.6	1D
8	CR	885	1 ½ hr.	920	1.2	2D
8	CR	885	45 min.	885	2.0	5D
12	CR	920	1 ½ hr.	885	1.8	1H
12	CR	920	1 ½ hr.	900	2.0	2H
12	CR	920	1 ½ hr.	920	0.5	3H
12	CR	920	1 ½ hr.	940	1.3	4H
22	CR	885	1 ½ hr.	885	1.4	1A
22	CR	885	1 ½ hr.	920	1.6	2A
22	CR	885	1 ½ hr.	825	Broke	3A
22	CR	885	45 min.	885	Broke	4A
8	U	920	1 ½ hr.	885	2.9	1J
8	U	920	1 ½ hr.	900	2.1	2J
8	U	920	1 ½ hr.	920	3.1	3J
8	U	920	1 ½ hr.	940	4.1	4J
12	U	920	1 ½ hr.	885	3.2	1F
12	U	920	1 ½ hr.	900	2.1	2F
12	U	920	1 ½ hr.	920	3.4	3F
12	U	920	1 ½ hr.	940	Off Center***	4F
16	U	920	1 ½ hr.	885	5.1	1I
16	U	920	1 ½ hr.	900	6.1	2I
16	U	920	1 ½ hr.	920	4.6	3I
16	U	920	1 ½ hr.	940	3.6	4I
12	U	750	1 ½ hr.	885	6.4	1B
12	U	750	1 ½ hr.	920	5.1	2B
12	U	750	1 ½ hr.	825	4.6	3B
12	U	750	45 min.	885	6.1	4B
12	U	825	1 ½ hr.	885	5.6	1C
12	U	825	1 ½ hr.	920	5.2	2C
12	U	825	1 ½ hr.	825	Broke	3C
12	U	825	45 min.	825	7.2	4C
12	U	885	1 ½ hr.	885	3.2	1E
12	U	885	1 ½ hr.	920	2.5	2E
12	U	885	1 ½ hr.	825	Broke	3E
12	U	885	45 min.	885	Broke	4E
12	U	920	1 ½ hr.	885	3.2	1F
12	U	920	1 ½ hr.	900	2.1	2F
12	U	920	1 ½ hr.	920	3.4	3F
12	U	920	1 ½ hr.	940	Off Center***	4F
12	U	950	1 ½ hr.	885	5.9	1G
12	U	950	1 ½ hr.	900	3.9	2G
12	U	950	1 ½ hr.	920	7.3	3G
12	U	950	1 ½ hr.	940	5.0	4G

*See Figure 2.

**Average for one rolling schedule.

***The part drawn was not centered in the blank, which prevented measuring the earing.

rolled conditions was 2.1%. Analysis 4 in Appendix A shows that it is statistically significant ($\alpha = 0.10$) that cross rolling reduced earing. This α value indicates there is a 10% chance that earing is not affected by cross rolling.

Table 5 also shows that during unidirectional rolling of vanadium, earing can be reduced by selecting the temperature of the initial anneal and rolling in fewer passes, i.e., heavier reductions per pass. Figure 10 portrays these observations. The blanks rolled in 8 or 12 passes had less earing in the formed parts than parts formed from sheet rolled in 16 passes. Also, the parts formed from blanks initially annealed at 885 and 920 °C had less earing than those formed from vanadium initially annealed at 750, 825, and 950 °C.

Analysis of earing data could not attach any statistical confidence to the observed influences of rolling pass schedule on earing, probably because of insufficient data. A quadratic model of initial anneal was fit to the percent earing data using least square regression. This model fitting approach was necessary because without duplicate samples, an independent error estimate approach was not possible. The results indicate that the model does not describe the data well; however, this does not imply that initial anneal temperature has no effect on earing.

The influence of the final anneal on earing is shown in Figure 10 (right side). The 885 and 920 °C final anneal temperatures show a pattern; i.e., with increasing initial anneal temperature, the earing increased for the product with the higher final anneal. This occurred because the two high-temperature anneals swung the texture balance toward annealing texture and the annealing ears increased. Statistical Analysis 1 (Appendix A) showed no significant influence of the final anneal averages on earing; however, synergistic effect between initial and final anneal is statistically significant at the $\alpha = 0.05$ level. The data analyzed are connected by lines in Figure 10. Looking beyond these data to the product with 950 °C initial anneal and 940 °C final anneal, the earing values should be greater than for the 920 °C final anneal because higher temperature annealing means more pronounced

Table 5 shows that earing can be substantially reduced by cross rolling. The three cross-rolling schedules produced parts with an average of 1.5% ears. The earing from the best unidirectionally

annealing texture. More data are required to determine whether this is an error in data point or that the trend is not real.

EARING DISCUSSION

Even though this experiment does not show a statistically or graphically illustrated influence of final anneal or earing, metallurgical logic says there must be some influence. The difference in earing between formed parts from sheet with the same rolling and initial anneal practice is primarily attributed to the final anneal the blanks receive before the forming operation.

The fact that this experiment showed a pattern for unidirectionally rolled sheet between earing results and initial anneal temperature is not surprising. Industry has demonstrated that the annealing temperature influences the annealing texture of metals and, thus, the earing.^{2,3}

But the influence of strain rate during rolling, i.e., reduction per pass, on earing apparently is not recorded in literature. If this phenomenon can be duplicated on larger rolling sections, then this experimental result would be significant for producing deep drawing quality (non-earring) metal.

The cross-rolled vanadium was all initially annealed at 885 and 920 °C, the two most favored temperatures, so data are not available indicating that initial anneal affects earing for cross-rolled vanadium. The severity of rolling reduction shows no influence on earing for cross-rolled plate because the strain rate is not cumulative when the direction of rolling changes on each pass.

CONCLUSIONS

The following conclusions were made using the results of this study:

1. The earing in formed vanadium parts can be improved by:
 - Producing the sheet with a cross-roll schedule where the plate is rotated 90° after each pass.

- Selecting the initial anneal (at the 1-inch thickness) between 885 and 920 °C (for 1 1/2 hours) if the vanadium is to be rolled uniaxially.
- Avoiding light rolling reductions per pass. In unidirectionally rolled product, a minimum reduction of 16% per pass is desired.
- The final anneals used within this experiment did not show a pattern of influence on the earing of the vanadium sheet.

2. The grain size of vanadium is primarily determined by the final anneal temperature. However, for unidirectionally rolled vanadium, the reduction schedule does have some influence. The grain size is finer on vanadium rolled in 8 to 12 passes than that rolled in 16 passes except when the final anneal temperature exceeds 900 °C and grains start to grow. With the heavier rolling reductions, grain growth occurs at a lower anneal temperature.

RECOMMENDATIONS

For the production of vanadium sheet where the metal is rolled primarily in one direction (unidirectional), the following fabricating procedure is recommended to produce sheet of minimum earing quality and grain size of ASTM 6 or finer:

- Anneal the 1-inch-thick plate for two hours between 885 and 920 °C before rolling to final thickness.
- Roll with a pass schedule providing 16 to 25% reduction per pass.
- Final anneal at a temperature and time providing fully recrystallized grains of ASTM 6 size or smaller. This temperature depends on the chemistry of the vanadium. For the vanadium used in this experiment, the best grain size was obtained when annealing 1 1/2 hours between 885 and 900 °C.

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ILLUSTRATIONS (Figures 1 through 10)

Note: Arrows show direction of rolling

FIGURE 1. Sections Cut for Rolling

FIGURE 2. Fabrication Tree

FIGURE 2. Fabrication Tree (Continued)

FIGURE 3. Punch

FIGURE 4. Draw Die

FIGURE 5. As-Rolled Vanadium at 0.125-in. Thickness

Section A - Annealed 1 1/2 hr at 885 °C
Cross Rolled 22 Passes

Section D - Annealed 1 1/2 hr at 885 °C
Cross Rolled 8 Passes

Section H - Annealed 2 hr at 920 °C
Cross Rolled 12 Passes

Section F - Annealed 2 hr at 920 °C
Unidirectionally Rolled 12 Passes

Section B - Annealed 1 1/2 hr at 750 °C
Unidirectionally Rolled 12 Passes

Section G - Annealed 2 hr at 950 °C
Unidirectionally Rolled 12 Passes

FIGURE 6. Grain Size Versus Final Anneal, 12 Unidirectional Rolling Passes

FIGURE 7. Grain Size Versus Final Anneal Influenced by Rolling Procedure

FIGURE 8. Photos Representing Grain Size Variation in Vanadium Produced

Sample 3A - ASTM 8 Grain Size
90% Recrystallized 100X

Sample 4E - ASTM 8 Grain Size
100% Recrystallized 100X

Sample 1H - ASTM 6-1/2-7-1/2 Grain Size 100X

Sample 3G - ASTM 5-1/2-6-1/2 Grain Size 100X

Sample 2I - ASTM 4-1/2 Grain Size 100X

Sample 4J - ASTM 3 Grain Size 100X

Part 2A, 1.6% Earing

Part 2B, 5.1% Earing

FIGURE 9. Earing Test Cups

FIGURE 10. Earing Versus Rolling Passes and Initial Anneal

APPENDIX A. STATISTICAL ANALYSIS*

Statisticians prefer that experiments be performed so that the interaction of all variables can be examined. To them, the variables include: (1) plate number, (2) temperature of initial anneal, (3) soak time of initial anneal, (4) rolling method (unidirectional or cross rolled), (5) number of passes, (6) temperature of final anneal, and (7) soak time of the final anneal. With this large number of independent variables, there are insufficient data points to make a complete interaction analysis.

With metallurgical logic, the following assumption can be made: First, plate numbers do not influence the earing and grain size results since both plates came from the same ingot and are adjacent cuts. Second, the difference between 1 1/2- and 2-hour soak times in the initial anneals can be ignored since the half-hour difference is insufficient to cause grain growth. These assumptions create the independent variable matrix shown in Table 1.

The empty cells in the matrix require that the statistical analysis be performed as a split-split plot.

*Performed by Barb Lawton, Statistical Quality Control and Analysis, Rockwell, Rocky Flats. Reported March 23, 1988.

TABLE 1. Overall Experiment Matrix

Roll	Final Anneal (°C)				Initial Anneal (°C)
	Roll Passes				
	8	12	16	22	
Unidirectional		825 885 885* 920			750
		825 885 885* 920			825
		825 885 885* 920			885
	885 920 900 940	885 920 900 940	885 920 900 940		920
		885 920 900 940			
Cross		885 920 900 940			950
	825 920* 885 920 885*			825 885 885* 920	885
		885 920 900 940			920

*Signifies a 45-minute anneal versus 1 1/2 hours for the other temperatures.

Four split-split plot analyses were performed. The first two analyses were performed using Tables 2a and 2b.

ANALYSIS 1

Objective

Examine the effect of final anneal temperature and determine whether there is a synergistic effect between final anneal temperature and pre-rolling anneal temperature.

Data

The data used for this analysis came from the interior of Tables 2a and 2b under Rows 885 and 920. All data in these tables came from the segments unidirectionally rolled with 12 passes. Data in the other final temperature rows were ignored since many of the cells were without values.

TABLE 2a. Percent Earing Values

Initial Anneal (°C)	Final Anneal Temperature (°C) (% Earing)					Avg. 885 & 920 °C (%)
	825	885	900	920	940	
750	4.6	6.4		5.1		5.75
825		5.6		5.2		5.4
885		3.2		2.5		2.85
920		3.2	2.1	3.4		3.3
950		5.9	3.9	7.3	5.0	6.6
	4.86			4.7		

TABLE 2b. Grain Size Values

Initial Anneal (°C)	Final Anneal Temperature (°C) (Grain Size)					Avg. 885 & 920 °C (%)
	825	885	900	920	940	
750	8.5	7.5		6.75		7.125
825	7.75	6.5		6.5		6.5
885	8.25	7.5		7.0		7.25
920		7.0	7.0	6.0	5.5	6.5
950		6.0	6.5	6.0	4.5	6.0
	6.9			6.0		

Results

1. On the average, there is no statistically significant change in either percent earing or grain size associated with a change in final anneal temperature from 885 to 920 °C. The lack of significance can mean one of two things: either final anneal temperature has no effect within the range examined, or there are too few observations to detect the effect.
2. There appears to be a statistically significant (at the $\alpha = 0.05$ level, i.e., 95% confidence level) synergistic effect between initial and final anneal temperatures for earing only. The effect is most easily seen by examining the difference in earing values between samples final annealed at 885 °C versus 920 °C with increasing initial anneal temperatures. At low initial annealing temperatures, earing can be reduced by final annealing at 920 °C, while at high initial annealing temperatures, earing can be reduced by final annealing at 885 °C. The probability of obtaining a pattern such as the one observed, assuming the null hypothesis of no synergistic effect is true (i.e., the p-value), is 0.03.
3. The data can be further divided into two subsets to provide additional insight into the effect of final anneal temperature when the sections are unidirectionally rolled with twelve passes. The first subset contains the values under the 825 final anneal temperature row and the second subset includes the values along 920 and 950 initial anneal temperature rows. Although neither of the subsets can be statistically evaluated (since there are no other samples prepared the same way to provide an error estimate), visual examinations of the data indicate that the final anneal temperature of 825 °C produces parts with consistently greater grain size values when the initial anneal temperatures are in the range of 750 to 885 °C. A final anneal temperature of 940 °C appears to decrease earing when the initial anneal temperatures are in the range of 920 to 950 °C. Since the majority of the percent earing values are missing for the additional samples examined here, no further information

is available regarding the effect of final anneal temperature on earing.

ANALYSIS 2

Objective

Examine the effect of the initial anneal temperature.

Data

The values used for this analysis come from Table 2a and 2b using the row averages found outside the matrix. The row averages are for the values found under the final anneal temperature of 885 and 920 °C.

Results

The statistical model used in analysis 2 assumes that effect of initial anneal temperature can be adequately described with a quadratic equation in temperature. The analysis proceeds by fitting a cubic equation in temperature and then using the cubic term to estimate the inherent variability or error in the data. Although the quality (and hence usefulness) of this error estimate is dubious, there is no other alternative. An independent estimate of process variability or error is normally obtained by evaluating the difference between multiple sections prepared in the same way. In this experiment, no two sections were prepared alike.

Based on the above analysis, initial anneal temperature does not have a statistically significant effect on either response variable within the range of temperatures examined. A visual examination of the data, however, tends to indicate a relationship between initial anneal temperature and earing. The most likely reason for this discrepancy is that the assumption of a quadratic model is invalid, which a plot of the earing values against temperature tends to support. The only way to determine whether initial anneal temperature does, in fact, affect the response variables is to obtain an independent

TABLE 3a. Percent Earing Values

		Final Anneal Temperature (°C)					Avg. 885 & 920 °C (%)
		825	885	900	920	940	
Unidirectional Rolling & Number of Passes	8		2.9	2.1	3.1	4.1	3.0
	12		3.2		2.5		2.85
	16		3.2	2.1	3.4		3.30
			5.1	6.1			4.85
Cross Rolling & Number of Passes (Actual Passes, 22)	8		1.6		1.2		1.4
	12		1.8	2.0	0.5	1.3	1.15
	16		1.4		1.6		1.5
			2.79		2.43		

TABLE 3b. Grain Size Values

		Final Anneal Temperature (°C)					Avg. 885 & 920 °C (%)
		825	885	900	920	940	
Unidirectional Rolling & Number of Passes	8		5.0	6.0	3.5	3.0	4.25
	12		7.5		7.0		7.25
	16	8.25	7.0	7.0	6.0	5.5	6.50
			4.5	4.5	4.5	5.0	4.85
Cross Rolling & Number of Passes (Actual Passes, 22)	8	7.8	7.25		6.75		7.00
	12		7.0	6.5	5.25	3.5	6.12
	16	8.0	7.25		6.5		6.87
			6.5		5.6		

Objective - Analysis 4

Examine the effect of rolling direction and rolling schedule.

Data

The data used for Analysis 3 came from the interior of Tables 3a and 3b under the final anneal temperature rows of 885 and 920 °C. The row averages outside the cell matrix represent the segment averages used in Analysis 4. All data in these tables came from segments that were initially annealed at either 885 or 920 °C. The data in the last row of the tables are actually from a section cross rolled with 22 passes rather than 16 passes.

Assumptions

Besides the general assumptions that changing plate numbers and varying the initial anneal time from 1 1/2 to 2 hours have no effect on earing and grain size, there were two other assumptions for Analyses 3 and 4. First, a change in initial annealing temperature from 885 to 920 °C does not affect either variable directly or indirectly with interactions between variables. Second, the effect on the response variables by cross rolling with 22 passes would be the same as if rolled in 16 passes.

Results

In the analysis of Tables 3a and 3b, the cells with two values are considered repeat samples. The difference between the average section values (found outside the cell matrix) is used to estimate the inherent variability (i.e., error of the rolling process) for evaluating the effect of rolling direction and rolling schedule.

1. The only factor having statistically significant effect (at the $\alpha = 0.10$ level) on earing is rolling direction. Whereas samples from unidirectionally rolled sections have an

error estimate by obtaining more data on samples prepared the same way.

The third and four analyses were made using Tables 3a and 3b.

ANALYSES 3 and 4

Objective - Analysis 3

Examine the effect of final anneal temperature and determine whether there is a synergistic effect between final anneal temperature and rolling direction and schedule.

average earing value of 3.5, those that have been cross-rolled have an average value of 1.4.

A visual examination of the data might suggest that the earing is greater when the part is unidirectionally rolled with 16 passes. The data for unidirectionally rolled samples with a final anneal temperature of 900 °C, although not included in the analysis, tend to follow the same pattern. This effect, if real, represents a two-factor interaction (i.e., synergistic effect) between rolling direction and final anneal temperature. Once again, the lack of statistical significance may be due to insufficient power (caused by too few observations) to detect effect of such small magnitude. More sections and samples produced according to a well-designed experimental plan are needed to determine whether this effect is real.

2. The only factor with a statistically significant effect (at the $\alpha = 0.10$ level) on grain size is final annealing temperature. On the average, the grain size value for samples final annealed at 920 °C is 5.6 whereas the average for samples annealed at 885 °C is 6.5. Although no other effects are statistically significant, an examination of the relative magnitude of the F statistics used to evaluate statistical significance and of the data displayed in Table 3b suggests that interaction between rolling direction and number of passes may also be important.

The average grain size values are similar for cross-rolled sections and sections rolled unidirectionally with 12 passes. The values for sections unidirectionally rolled with either

8 or 16 passes are similar to one another, but substantially lower than the values in the preceding group. This pattern holds not just for those samples used in the formal statistical analysis, but for all samples displayed in the table. Once again, more sections and samples produced in accordance with a well-designed experiment are needed to determine whether this effect is real.

DISCUSSION

The optimal condition for producing drawn parts (i.e., the percent earing is minimized and the grain size measure is maximized) is to cross roll the section and to final anneal at temperatures in the range 825 to 885 °C. The number of passes for cross rolling used does not appear to have much impact on earing and grain size, and the effect of initial annealing time and temperature is unknown.

If sections must be rolled unidirectionally, then the optimum condition is 12 passes, with a pre-rolling annealing temperature in the range of 885 to 920 °C. Sections prepared this way have yielded samples with only slightly higher earing values than cross-rolled sheet and comparable grain size values. The effect of final anneal temperature is similar to that for cross-rolled parts.

More than half the data obtained in this experiment could not be statistically analyzed due to the nature of the experimental plan used. If statistical analysis of the data is desired, then the principles of experimental design and statistical evaluation of the data generated should be considered in the planning stage of the experiment. This will ensure that the data collected yield the desired information.