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RECRYSTALLIZATION OF BETA-HEAT-TREATED
DINGOT-URANIUM CORES

August 24, 1962

WELDON SPRING PLANT

MALLINCKRODT CHEMICAL WORKS
URANIUM DIVISION

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TOPICAL REPORT

RECRYSTALLIZATION OF BETA-HEAT-TREATED DINGOT-URANIUM CORES

by

R. R. Guyer
N. F. Neumann

August 24, 1962

Mallinckrodt Chemical Works
Uranium Division
Weldon Spring, Missouri

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RECRYSTALLIZATION OF BETA-HEAT-TREATED DINGOT-URANIUM CORES

by

R. R. Guyer
N. F. Neumann

A B S T R A C T

Annealing beta-heat-treated production-size dingot-uranium core blanks in the high alpha range (1150-1200°F) produced a fine, equiaxed grain size by a recrystallization process. The extent of recrystallization is dependent upon the rate of cooling through the beta-to-alpha transformation, the time and temperature of annealing, and whether or not impurities that retard grain growth are present. The cooling rate through the beta-to-alpha transformation was varied by using a range of beta temperatures and air cooling times prior to quenching into water. Longer air cooling times are permissible at the higher beta temperatures; however, the greatest amount of induced lattice strain was obtained after an air delay of only 2 seconds before quenching. Decreasing the annealing temperature results in a corresponding increase in the annealing time required for an equivalent amount of recrystallization. A second phase, present in the alloyed dingot metal, inhibited recrystallization and grain growth. Alloyed metal recrystallized progressively from the outer periphery to the center of the core, whereas unalloyed metal subjected to identical annealing conditions exhibited recrystallized grains throughout the cross section. A reduction in grain size from 0.38 mm to 0.25 mm occurred in a beta-treated, unalloyed core after annealing at 1200°F for 4 hours. The grain size of beta-quenched, alloyed metal decreased from 0.28 mm to 0.19 mm on recrystallization. There appears to be no change in the orientation of a beta-quenched core as a result of recrystallization. The beta quench-alpha anneal process could be integrated with an alpha-phase vacuum outgassing process to produce fine-grained metal that is essentially free of strain and preferred orientation and has a low hydrogen content.

RECRYSTALLIZATION OF BETA-HEAT-TREATED DINGOT-URANIUM CORES

by

R. R. Guyer*

N. F. Neumann

INTRODUCTION

Massive dingots of uranium are extruded in the gamma phase (1850°F) to form a bar 6.5 to 7 inches in diameter and 8 to 10 feet long. Billets are cut from the extruded bar for subsequent alpha-phase (1175°F) rolling or extrusion to produce a 1.5-inch-diameter rod. Core blanks (approximately 8.5 inches long) are cut from the rods. The alpha-worked microstructure (see Fig. 1) is normally very fine. However, the as-rolled or alpha-extruded rod is highly oriented in the direction of working and has the tendency to grow during irradiation and thermal cycling.¹

Heat treatment in the beta phase essentially eliminates the preferred orientation normally present in alpha-worked structure. Time and temperature within the beta phase are not critical, except for the need to allow adequate time for complete transformation. Slow cooling through the beta-to-alpha phase transformation will produce a structure having large, irregularly shaped grains with numerous twins and subgrains. If the cooling from the beta phase is rapid, however, the grain size will be finer. The macro grain structure associated with the latter type of beta heat treatment is shown in Fig. 2.

A coarse grain size is believed to cause a condition during irradiation known as "surface roughening."² Surface roughening can be eliminated by increasing the cladding thickness or by reducing the grain size of the fuel element.³ Refinement of the grain structure is the more desirable solution since increasing the cladding thickness would produce a corresponding decrease in reactivity.³

Previous work at Mallinckrodt⁴ indicated no appreciable grain refinement was obtained in unalloyed dingot-uranium cores by varying the temperature of beta heat treatment, the time at temperature (beyond that required for complete transformation), and the cooling time in air prior to water-quenching.

Grain refinement has been obtained in beta-quenched uranium by the addition of small amounts of alloying elements such as iron,⁵ silicon and chromium.^{6,7} These elements have limited solubility in alpha uranium. When the solubility is exceeded they form compounds with the uranium which precipitate from solution. Such second phases inhibit grain growth during subsequent thermal treatment.⁸

* Present address: Lockheed Aircraft Corporation, Marietta, Georgia.

Fig. 1. Typical Microstructure of Alpha-Rolled Dingot Uranium.
Electropolished, polarized light, 100X magnification.

Fig. 2. Typical Structure of Beta-Treated Dingot Uranium.

Beta treatment: 1350°F for 15 minutes; 43-second air delay, quench in 130°F brine. Standard HCl, HNO₃ macroetch; bright field illumination; 30X magnification.

A second method of producing a fine grain size in a fuel element, other than by alloy additions, would be by recrystallization of the beta-quenched structure. Alpha annealing of beta-treated and water-quenched uranium will produce a fine, equiaxed grain structure resembling that obtained by recrystallization of alpha-worked uranium (see Fig. 3).

The work by Gardner and Riches⁹ indicated that the extent of recrystallization during alpha annealing was dependent on the cooling rate from the beta phase. Furthermore, the activation energy for recrystallization was found to be inversely related to the cooling rate.

The presence of quench-induced strain within the alpha lattice has been demonstrated by broad X-ray diffraction lines that become sharper after the annealing treatment.¹⁰ Evidently, this strain is sufficient to result in recrystallization during subsequent alpha annealing. The lattice strain is apparently caused by the large volume change that occurs during transformation, coupled with the pronounced anisotropic nature of the thermal contraction of alpha uranium.

In this report, the feasibility of the beta quench-alpha anneal process as a method to refine the grain structure of beta-treated ingot-uranium cores is discussed. The effect of time and temperature of annealing, amount of alloying elements, and heat-treating variables on the kinetics of recrystallization are reported. Some of these parameters have been investigated with ingot-uranium cores at the Savannah River Laboratory by Angerman, Huntoon, and McDonnell.¹¹

EXPERIMENTAL PROCEDURE

Full-size solid and hollow core blanks from four normal-production alloyed ingots and two unalloyed ingots (see Table I for chemical analysis) were used in the recrystallization studies. The core blanks were machined from 1.5-inch-diameter alpha-rolled rods to a 1.433-inch OD and cut to an 8-1/2-inch length. The hollow cores were made by drilling a 0.416-inch-diameter hole in the solid blank.

The samples were beta-heat-treated in a laboratory salt bath composed of a 50 w/o NaCl - 50 w/o KCl mixture at 1300°, 1350°, and 1400°F for 15 minutes. Then they were air-cooled for 2, 10, 45, and 60 seconds prior to quenching in 60°F or 130°F water. These ranges of beta temperature, delay time, and quench water temperature were selected to give a variety of quench rates.

To remove that portion of the metal containing end-effects from quenching and to provide specimens for study of the beta-quenched structure, a 3/4-inch-long section was cut from both ends of each beta-treated core blank. The cut faces of these pieces were etched, first in concentrated hydrochloric acid and then in concentrated nitric acid, and then were photographed at 3X magnification. Evaluation of the macrostructure was made from the photographs.

The remainder of each beta-treated core was sectioned into 1-1/2-inch lengths for annealing. Alpha annealing was done for various lengths of time in a molten carbonate bath at temperatures from 950°F through 1200°F. For times at temperatures in excess of 4 hours the core sections were protected from the salt by placing them in a steel vessel. To prevent oxidation, a positive pressure of helium was maintained in the vessel during the annealing cycle. After the prescribed annealing time, the vessel and its enclosed sample were removed from the salt bath and were water-cooled to room temperature.

TRANSVERSE SECTION

LONGITUDINAL SECTION

Fig. 3. Typical Structures of Beta-Treated, Alpha-Annealed Alloyed DIngot Uranium.
 Beta treatment: 15 minutes at 1350°F, 2-second air delay, quench in 130°F brine.
 Anneal: 16 hours at 1200°F.
 Standard HCl, HNO₃ macroetch; bright field illumination; 20X magnification.

Table I

Concentration of Some Elements in Dingot Uranium

Element	Concentration ppm						
	Alloyed Dingot Number					Unalloyed Dingot Number	
	2168	5144	5327	5522	3159	1629	4722
Al	50.	32.	17.	14.	< 10.	-	18.
Cr	20.	9.	5.	6.	< 5.	< 5.	12.
Cu	> 10.	5.	3.	2.	< 5.	-	6.
Ni	45.	40.	20.	14.	12.	12.	50.
Fe	110.	164.	141.	134.	150.	48.	35.
Si	150.	91.	90.	94.	100.	18.	16.
N ₂	16.	36.	21.	17.	16.	6.	6.
H ₂	1.4	2.3	2.9	-	2.7	2.3	2.9
Mg	15.	< 10.	< 10.	< 10.	< 10.	14.	10.
Mn	14.	< 10.	< 10.	< 10.	< 10.	< 10.	< 10.
C	-	20.	21.	31.	-	-	-

The annealed samples were cut in half transversely, and the cut surfaces were etched in concentrated HCl and HNO₃, and examined. For determination of the extent of recrystallization, a line was drawn around the unrecrystallized areas while observing the structure at 30X magnification. The area of recrystallization was then measured with a polar planimeter, and the percentage of recrystallized metal was calculated. In some cases, estimates were made of the extent of recrystallization by comparing the inscribed unrecrystallized area with a transparent overlay composed of circles representing 5 percent increments of recrystallization.

RESULTS AND DISCUSSION

The variation in the percentage of recrystallized metal with alpha annealing time and temperature is shown in Fig. 4 for alloyed and unalloyed dingot-uranium cores, solid and I&E.* The "S" shape of the curves is typical of curves obtained for the recrystallization of cold-worked metal. Lowering the annealing temperature causes a corresponding shift of the curve to the right; i.e., requires an increased time of annealing for equivalent amounts of recrystallization.

Photomacrographs of annealed, alloyed dingot-uranium solid-core samples show the effect of annealing temperature (Fig. 5) and time (Fig. 6) on recrystallization. Raising the annealing temperature from 1080°F to 1200°F and holding at temperature for 2 hours resulted in an increase in the recrystallization from approximately 10 percent to approximately 98 percent. A similar increase in recrystallization was obtained by extending the annealing time from 5 minutes to 120 minutes at 1200°F.

The effect of dingot composition on the extent of recrystallization is shown in Exhibit B in Fig. 7. The alloyed uranium core, which contained approximately 150 ppm iron and 100 ppm silicon, was 76 percent recrystallized after 30 minutes at 1200°F

* Internally and externally cooled (hollow) cores.

ALLOYED URANIUM SOLID CORE BLANKS

UNALLOYED URANIUM SOLID CORE BLANKS

ALLOYED URANIUM HOLLOW CORE BLANKS

UNALLOYED URANIUM HOLLOW CORE BLANKS

Fig. 4. The Effect of Time and Temperature of Alpha Annealing on the Recrystallization of Beta-Heat-Treated Core Blanks. Beta treatment: 15 minutes at 1350°F, 2-second air delay, quench in 60°F water.

Alloyed uranium: iron content = 150 ppm and silicon content = 100 ppm.
 Unalloyed uranium: iron content = 48 ppm and silicon content = 18 ppm.

ANNEALED 2 HOURS AT GIVEN TEMPERATURE

Fig. 5. Photomicrographs Showing the Effect of Alpha Annealing Temperature of the Recrystallization of Beta-Heat-Treated, Alloyed Dingot-Uranium Solid Cores. Beta heat treatment conditions: held at 1350°F for 15 minutes; 2-second air delay; quenched in 60°F water. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

Fig. 6. Photomicrographs Showing the Effect of Time at Alpha Annealing Temperature on the Recrystallization of Beta-Heat-Treated, Alloyed Dingot-Uranium Solid Cores. Beta heat treatment conditions: held at 1350°F for 15 minutes; 2-second air delay; quenched in 60°F water. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

whereas the metal containing 48 ppm iron and 18 ppm silicon treated under the same conditions was 98 percent recrystallized.

The variation in the chemical composition of the alloyed dingots also influenced the time required for recrystallization. Dingots 5327 and 5522, which were nearly identical in their iron (141 ppm vs. 134 ppm) and silicon (90 ppm vs. 94 ppm) contents, had similar recrystallization characteristics. However, Dingot 5144, which contained 164 ppm iron and 91 ppm silicon, required approximately twice the amount of time at temperature for equal amounts of recrystallization. This effect is shown in Fig. 8 and Table II.

Recrystallization of alloyed metal begins at the outer periphery and proceeds toward the center of the core. Thus, in solid cores, recrystallization begins in the zone with the greatest cooling rate through the beta-to-alpha transformation. One might assume, therefore, that hollow cores would begin to recrystallize from both the outer and the inner surfaces. However, this did not always occur. Samples cut from the ends of such cores started to recrystallize at both the inner and outer surfaces while the ID surface was the last to recrystallize on pieces cut midway along the length of the core blank. This difference in the mode of recrystallization suggests that there was insufficient cooling of the inner surface during the beta quench, probably because of the formation of steam pockets that reduced the transfer of heat in these regions. In unalloyed cores the recrystallization appeared first at the outer periphery, but after a short time, small areas of recrystallized grains were observed throughout the cross section.

The cooling rate through the transformation was also varied by quenching from 1300°F, 1350°F, and 1400°F. With a constant air delay time of 45 seconds prior to quenching, cores from Dingot 5522 (134 ppm Fe and 94 ppm Si), beta-treated at 1400°F, were 95 percent recrystallized after annealing at 1200°F for 8 hours while cores from the same dingot beta-treated at 1300°F and given an identical anneal were only 85 percent recrystallized (see Table II).

Extending the air delay to a time when the core has transformed or is in the process of transformation produces a large-grained "feathery" structure. Such a structure was obtained when cores were air-cooled from 1300°F for 60 seconds before quenching (see Exhibit A in Fig. 9). These large-grained areas did not recrystallize during subsequent annealing; however, the small beta-quenched grains within the same core did recrystallize. This effect is shown in Exhibit B in Fig. 9.

These data show that decreasing the cooling rate through the transformation retards recrystallization and that a critical cooling rate exists below which recrystallization will not take place. The activation energy required for the recrystallization was calculated by means of the Arrhenius reaction rate equation, which was used in its logarithmic form,

$$\log \frac{t_2}{t_1} = \frac{Q}{2.303 R} \left[\frac{1}{T_2} - \frac{1}{T_1} \right],$$

where t_1 and t_2 are the times required for 50 percent recrystallization at absolute temperatures T_1 and T_2 , R is the gas constant in calories per mole per °K,

A

ALLOYED UNALLOYED

BETA-TREATED AT 1350°F FOR 15 MINUTES; 2-SECOND
AIR DELAY; QUENCHED IN 60°F WATER

B

ALLOYED
76% RECRYSTALLIZED

UNALLOYED
98% RECRYSTALLIZED

ALPHA-ANNEALED AT 1200°F FOR
30 MINUTES ; WATER-COOLED

Fig. 7. Photomicrographs Showing the Effect of Alloy Content on the Macrostructure of Beta-Heat-Treated, Alpha-Annealed Dingot-Uranium Solid Cores. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

Fig. 8. Relationship between Annealing Time and Temperature for Initiation and Completion of Recrystallization of Beta-Heat-Treated, Alloyed Dingot-Uranium Solid-Core Blanks. Beta heat treatment: held at 1350°F for 15 minutes; 10-second air delay; quenched in 130°F water.

Table II

Relationships Between the Beta Heat Treatment Conditions,
Alpha Annealing Conditions, and the Recrystallization

Dingot No.	Annealing Conditions		Estimated Percent Recrystallized							
	Temp. °F	Time hr	Beta Temperature and Air Delay Time							
			1300°F		1350°F			1400°F		
			10 sec	45 sec	10 sec	45 sec	60 sec	10 sec	45 sec	60 sec
5327	1200	8	90-95	90	95-100	90	90	95-100	90	85
5522		8	90-95	85	95-100	95	80	95	95	80
5144		16	90-95	75	95	85	85	95	90	90
5327	1150	32	90	80	95	85-90	90	95	90-95	90-95
5522		32	85-90	75	95	85	80	95-100	95-100	95-100
5144		64	90	75	95	85-90	80-85	90-95	90-95	80-85
5327	1100	128	80	80	85	80-85	80-85	90	90-95	85
5522		128	80	80	95	85	80	95-100	95	90-95
5144		256	75	70	85	85	80	85	85	90
5327	1100	2	< 5	< 5	5	< 5	< 5	5-10	5	5-10
5522		2	< 5	< 5	5	< 5	< 5	5-10	< 5	< 5
5144		4	5	< 5	5	5	5	5-10	5-10	5-10
5327	1050	8	5	< 5	5	< 5	< 5	5	5	5
5522		8	5	< 5	5	< 5	< 5	5	< 5	< 5
5144		16	< 5	0	< 5	0	0	< 5	< 5	0
5327	1000	64	5-10	< 5	5-10	< 5	< 5	5-10	5	< 5
5522		64	< 5	< 5	5-10	< 5	< 5	5-10	< 5	< 5
5144		128	< 5	< 5	5-10	< 5	< 5	5-10	< 5	< 5
5327	950	512	-	0	0	-	0	-	-	-
5522		512	-	0	0	-	0	-	-	-
5144		512	-	0	0	-	0	-	-	-

A

BETA-TREATED AT 1300°F FOR 15 MINUTES; 60-SECOND
AIR DELAY; QUENCHED IN 130°F WATER

B

ALPHA-ANNEALED AT 1200°F FOR
16 HOURS; WATER-COOLED

Fig. 9. Photomicrographs Showing the Effect of Low Beta Temperature and Long Air Delay on the Heat-Treated and Annealed Grain Structures of Alloyed Dingot-Uranium Solid Cores. The arrows indicate areas which contained "feathery" grains that did not recrystallize. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

and Q is the activation energy in calories per mole. A plot was made of the logarithm of the time required for 50 percent recrystallization as a function of the reciprocal of the absolute temperature of annealing. The activation energies for three different iron and silicon alloy levels were calculated from the slope of the curves in Fig. 10. Unalloyed (48 ppm Fe and 18 ppm Si) solid dingot-uranium cores had activation energies of 83,000 cal/mole. Still higher values of 89,000 cal/mole and 114,000 cal/mole were obtained for alloyed uranium from Dingots 3159 (150 ppm Fe and 100 ppm Si) and 2168 (110 ppm Fe and 150 ppm Si), respectively. Similar curves were plotted and activation energies calculated for the hollow cores. Metal from Dingot 3159 had identical activation energies for both solid and hollow cores; however, hollow cores from unalloyed Dingot 1629 had an activation energy of 67,000 cal/mole, which is considerably lower than that obtained for the solid cores from the same dingot. The accuracy with which the activation energies for the latter dingot could be determined was low. This resulted from difficulties in measuring the extent of recrystallization, since cores from this dingot recrystallized in random areas throughout the cross section.

The activation energies for recrystallization could also have been calculated from the curves in Fig. 8. However, inaccuracies associated with determination of the times required for 5 percent and 95 percent recrystallization do not warrant these calculations.

Measurements were made of the recrystallized grain size of alloyed and unalloyed dingot-uranium cores (see Figs. 11 and 12). As shown in Table III, a finer grain size resulted from the use of a high alpha temperature with a short annealing time as compared with the results for lower alpha temperatures with longer annealing times.

Work done at the Savannah River Laboratories¹² suggests that the change in structure proceeds not only by nucleation and growth of new grains but also by the growth of small grains that are present in the as-quenched structure. At the low annealing temperatures it is possible that fewer new grains are nucleated in a given length of time while at the same time the beta-quenched grains may have been growing. If this is the case, then the final grain size must depend on the number of nuclei, the rate of grain growth, and the amount and dispersion of any second phase that might retard grain growth. The grain size of beta-quenched, unalloyed metal decreased from 0.38 mm to 0.25 mm, on an average, through recrystallization at 1200°F, while a change from 0.28 mm to 0.19 mm occurred on recrystallization of alloyed dingot-uranium cores (Dingot 5327, Table III).

The structures produced by the various beta treatments were also examined. Acceptable grain structures were observed in all but one of the alloyed cores that were beta-heat-treated at 1300°F, 1350°F, and 1400°F and then given air cooling times of 10, 45, and 60 seconds prior to quenching into 130°F water. The exception was the sample with the 60-second delay after heating at 1300°F, which had the "feathery" structure described above. Unalloyed dingot-uranium core blanks, beta-treated at 1350°F for 15 minutes, then air-cooled 10, 20, and 40 seconds before quenching, had coarse grain structures. Typical photomicrographs of beta-treated dingot-uranium cores, alloyed and unalloyed, are shown in Exhibit A in Fig. 7. Increasing the air delay time before quenching results in a slight coarsening of the grain size. However, no consistent correlation was observed between the beta temperature value and the grain size of the beta-quenched metal.

Fig. 10. Relationship between Annealing Temperature and Time for 50 Percent Recrystallization of Beta-Heat-Treated, Alloyed and Unalloyed Dingot-Uranium Solid-Core Blanks. Beta heat treatment conditions: held at 1350°F for 15 minutes; 2-second air delay; quenched in 60°F water.

AS BETA-TREATED

1200°F - 4 HOURS

1150°F - 16 HOURS

1100°F - 64 HOURS

Fig. 11. Photomicrographs Showing the Effect of Time and Temperature of Alpha Annealing on the Recrystallized Macrostructure of Alloyed Dingot-Uranium Cores. Beta heat treatment conditions: held at 1350°F for 15 minutes; 10-second air delay; quenched in 130°F water. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

Fig. 12. Photomicrographs Showing the Effect of Time and Temperature of Alpha Annealing on the Recrystallized Macrostructure of Unalloyed Dingot-Uranium Cores. All of the annealed structures are fully recrystallized. Beta heat treatment conditions: held at 1350°F for 15 minutes; 10-second air delay; quenched in 130°F water. Standard HCl, HNO₃ macroetch; bright field illumination; 3X magnification.

Table III

Grain Size of Beta-Heat-Treated^a and Recrystallized Core Blanks

Diameter of Average Beta-Quenched Grain ^b		
mm		
Alloyed Dingt		Unalloyed Dingt
<u>5144</u>	<u>5327</u>	<u>4722</u>
0.25	0.28	0.38

Annealing Conditions		Diameter of Average Recrystallized Grain ^b		
mm		mm		
Temp. °F	Time hr	Alloyed Dingt		Unalloyed Dingt
		<u>5144</u>	<u>5327</u>	<u>4722</u>
1200	2	-	-	0.23
	4	-	0.19	.25
	8	0.17	.19	-
	16	.17	-	-
1150	8	-	-	.24
	16	-	.19	.25
	32	.17	.19	-
	64	.23	-	-
1100	32	-	-	.26
	64	-	.22	.31
	128	.24	.24	-
	256	.26	-	-

^a

Beta heat-treated for 15-minutes at 1350°F, then air-cooled for 10 seconds prior to quenching in 190°F water.

^b

Average grain diameters were calculated from Heyn line-intercept measurements. A 4-inch cross was drawn on 20X magnification photomicrographs at both OD and ID surfaces of hollow cores.

CONCLUSIONS

A fine, equiaxed grain structure is produced in uranium cores by the beta quench-alpha anneal process. The rate of recrystallization and the grain size of the recrystallized structure are dependent upon the cooling rate from the beta phase, the time and temperature of annealing, and the chemical composition. Sufficient strain is imparted to the uranium lattice during rapid cooling from the beta phase for subsequent recrystallization, especially if the air cooling time before quenching is less than 10 seconds. Recrystallization is complete in a shorter time and the grain size is finer when annealing is done in the high alpha range (1150° - 1200°F).

APPLICATION

One application of the beta-quench, alpha-anneal process would be vacuum outgassing in the alpha temperature range (for hydrogen control). Outgassing after a beta treatment would remove the hydrogen initially contained in the metal plus that picked up from the beta salt bath. Simultaneously it would produce a fine grain structure. Table II also indicates the times and temperatures that can be used to outgas beta-treated cores without recrystallization if this is desired.

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