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SOME ASPECTS OF THE BETA TO ALPHA
TRANSFORMATION IN URANIUM:
METALLOGRAPHIC STRUCTURE AND
ORIENTATION RELATIONSHIPS

by

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SOME ASPECTS OF THE BETA TO ALPHA TRANSFORMATION IN URANIUM: METALLOGRAPHIC STRUCTURE AND ORIENTATION RELATIONSHIPS

by
H. H. Chiswik and L. T. Lloyd

I. INTRODUCTION

Polycrystalline metals that undergo allotropic transformations on cooling from high temperatures frequently exhibit a feature of metallographic structure known as subgraining, which consists of a subdivision of the grains into smaller units of structure or "subgrains." A familiar example of such substructure is "veining" in alpha iron which is formed during the gamma to alpha transformation.⁽¹⁾ The occurrence of the substructures has been related by many investigators to the volume changes involved in the allotropic transformation, and the extent of their presence has been shown to be a function of cooling rate.

In uranium, subgraining is present when the metal is cooled from either beta-phase or gamma-phase temperatures. The subgrains are more readily detectable than in alpha iron because metallographic examination is usually made under polarized light, which delineates clearly the slight differences in the orientations between the subgrains. The occurrence of subgraining during the $\beta \rightarrow \alpha$ transformation has been one of the major difficulties responsible for the lack of success in utilizing the phase-transformation technique for growing single crystals of alpha uranium.⁽²⁾ This report summarizes some observations on the effects of cooling rates and temperature gradients during the beta to alpha transformation on substructure and orientation relationships.

II. EFFECTS OF COOLING RATE

Microstructures of uranium were examined after a) water quenching; b) air cooling; c) furnace cooling at 4°C per hour; and d) furnace cooling at 0.5°C per hour.

For furnace cooling, the specimens were approximately 1/8 to 1/4 inch square in cross section by 1/2 inch long, and were cut from directionally solidified 1-inch diameter, electrolytic high-purity uranium castings. The specimens were encapsulated under vacuum in Vycor tubes and were heated at beta or gamma-phase temperatures in a furnace with a 2-inch central uniform temperature zone. Controlled cooling rates were obtained by the mechanical movement of the indicator pointer of an "off-on" temperature controller activated by a thermocouple located near the specimen at the center of the uniform temperature zone.

A. Air Cooling and Water Quenching

Typical microstructures of specimens annealed for 24 hours at 740°C and either air cooled or water quenched are shown in Figure 1. Unlike the slowly cooled specimens discussed below, all the grains in the water-quenched and air-cooled specimens showed evidences of subgraining. The subgrains appeared smaller and more regularly oriented with respect to the parent alpha grain than those in the slowly cooled specimens. A wide variation was noted in the sizes of the primary alpha grains* (compare grains A and B in Figure 1).

B. Furnace Cooling

The results of cooling at 4°C per hour from 800 to 600°C followed by air cooling to room temperature are illustrated in Figure 2. The treatment produced relatively large grains, some of them 3 to 4 mm in cross section, with sharp boundaries between them, in contrast to the irregular and jagged boundaries in water-quenched specimens. The majority of the grains in each specimen showed no microscopic evidence of subgraining (see grains A and B in Figure 2, and grain C in Figure 3). However, along with the relatively homogeneous grains there were present in each specimen several grains that contained a subgrained structure illustrated by grains D and E in Figures 2 and 3. Cooling at the same rate from beta-phase temperatures (about 740°C), or varying the annealing time in the beta range from 2 hours to 2 weeks, had no effect on the microstructure (Figure 3).

Back-reflection X-ray Laue photograms from the grains of the slowly cooled specimens are illustrated in Figures 4 and 5. For comparison, a Laue photogram was also taken under identical conditions from a large grain of a polycrystalline specimen that had been critically strained and recrystallized in the alpha phase (Figures 4 and 6; the Debye rings are from the thin lead shield placed over the specimen to line up the beam on the grain). The reflection spots from the microscopically homogeneous grain A in Figure 2 are relatively sharp and distinct; when compared with the reflections from the alpha recrystallized grain, however, they show a slight tendency to be radially elongated, suggesting the presence of a strain in the lattice that could not be detected microscopically. The photogram from the microscopically subdivided grain D in Figure 2 is what one might expect from a grain that has broken up into a large number of subgrains differing slightly in orientation from each other.

*Grains separated by high angle boundaries

Figure 1. High-purity Uranium Water Quenched from 740°C.
Polarized Light.

a. 0° Stage Rotation

11,825

50X

b. 45° Stage Rotation

11,826

50X

Figure 2. High-purity Uranium Cooled at 4°C per Hour from 800 to 600°C Followed by Air Cooling to Room Temperature. Polarized Light.

a. 0° Stage Rotation

11,832

50X

b. 45° Stage Rotation

11,829

50X

Figure 3. High-purity Uranium Cooled at 4°C Per Hour from 740°C . Polarized Light.

a. 0° Stage Rotation

b. 45° Stage Rotation

Figure 4. Back-reflection Laue Photogram from Microscopically Homogeneous Grain A in Figure 2. (Note: Debye rings are from thin lead shield placed over the specimen to line up the beam on the grain.)

11,062

Figure 5. Back-reflection Laue Photogram from Microscopically Subdivided Grain D in Figure 2.

11,064

Figure 6. X-ray Back-reflection Laue Photogram from a Large Alpha Grain in a Cold-worked and Recrystallized Specimen. (Note: Debye rings are from thin lead shield placed over the specimen to line up the beam on the grain.)

11,065

The extent of subgraining was further reduced by lowering the cooling rate to 0.5°C per hour. In three out of five specimens cooled at this rate from 800 or 740 to 600°C and subsequently air cooled to room temperature, all the grains were completely free from microscopic subgraining; the other two specimens showed evidences of subgraining in only a few of the peripheral grains. In each of the specimens there was at least one microscopically homogeneous grain that extended through the entire cross section of the specimen ($1/8$ inch on a side). A typical example of such a grain is shown in Figure 7. In a few instances some of the large grains contained one or two low-angle boundaries, as evidenced by slight discontinuities in the twin directions (Figure 8). By comparison with the specimens cooled at 4°C per hour, it is evident that the further reduction in the cooling rate has considerably reduced the tendency to subgraining, but has not completely eliminated it.

Figure 7. High-purity Uranium Specimen Cooled at 0.5°C per Hour from 800°C

11,948

25X

Figure 8. High-purity Uranium Specimen Cooled at 0.5°C per Hour from 800°C

11,949

5X

III. ORIENTATION RELATIONSHIPS

The crystallographic orientations of the alpha grains obtained by slow cooling from the beta phase under imposed temperature gradients were determined for three groups of samples. The specimens were of high purity with a total impurity content of less than 100 parts per million (by weight). The calculated cooling rates at the beta-to-alpha transformation temperatures were about 0.5°C per hour, and the temperature gradients at the transformation temperature ranged from 1.3 to 138°C per inch (see Table I). The orientations were determined from back-reflection Laue photograms obtained with the aid of a micrometer X-ray specimen holder.⁽³⁾

TABLE I

Time and Temperature Conditions of Beta to Alpha Transformation Treatments

Specimen Number	Rate of Cooling at Beta to Alpha Transformation Temperature ($^{\circ}\text{C}/\text{hour}$)	Temperature Gradient at Beta to Alpha Transformation Temperature ($^{\circ}\text{C}/\text{inch}$)	Rate of Passage of Beta to Alpha Transformation Interface Along Length of Specimen (inches/day)	Duration of Test (days)
5-16-52-1	0.47	138	0.082	39
5-16-52-2	0.47	138	0.082	39
5-16-52-3	0.47	138	0.082	39
6-12-52-1	0.49	117*	0.101	27.5
6-12-52-2	0.57	109*	0.125	18
6-12-52-3	0.56	96*	0.140	17
B272-1	0.50	1.3	9.230	9
B272-2	0.50	1.3	9.230	9
B272-3	0.50	1.3	9.230	9
B272-4	0.50	1.3	9.230	9
B272-5	0.50	1.3	9.230	9

*These gradients were measured in the furnace. The gradients in the encapsulated samples undoubtedly were somewhat smaller.

A. Specimens 5-16-52-1, -2 and -3

The specimens were prepared by machining 300°C swaged rods into "nails" with heads 0.148 inch in diameter by 0.125 inch long, bodies 0.115 inch in diameter by approximately $3/4$ inch long, and points with a taper of $1/4$ inch per inch. The cooling was carried out in the temperature gradient of the NaK-filled, vertical tube furnace.⁽²⁾ The specimens were suspended by their heads from a wire-supported tantalum disc. The assembly was placed in the furnace at a temperature within the beta

phase and then lowered, by means of the wire and a slow speed motor, through the temperature gradient into a zone within the alpha-phase temperature range. Because the supporting wires broke during a prior run, the structure of these samples before the slow cooling treatment was not that of 300°C swaged material, but corresponded to that obtained in a moderately rapid quench from the beta phase. The temperature gradient attained in these specimens was 138°C per inch.

In Figure 9 are shown line tracings of the grain structures of these specimens on longitudinal sections.* As indicated by the dotted lines, a large degree of subgraining was present. The grains whose orientations were determined are designated by numbers, and in Table II are given the angles between the principal crystallographic axes of each

Figure 9. Line Tracings of Grain Boundaries on Longitudinal Section of Specimens 5-16-52-1, -2 and -3. Solid lines represent high angle grain boundaries; dotted lines represent subgrain boundaries. Numbers designate areas whose orientations were determined by Laue back-reflection X-ray photograms.

a. Specimen 5-16-52-1

b. Specimen 5-16-52-2

c. Specimen 5-16-52-3

22,435

3X

* Line tracings rather than photomicrographs are shown for these and for some of the other specimens because the subgrain structure did not show up well when the composite photographs were reduced in size.

TABLE II

Orientations of Grains Relative to Direction of Temperature Gradient

Specimens 5-16-52-1, -2, -3, 6-12-52-1, -2 and -3						Specimens B272-1 bottom, -1 tip, -2 tip and -4 tip					
Specimen No.	Grain Designation		Angle between Direction of Temperature Gradient and Principal Plane Pole - (degrees)			Specimen No.	Grain Designation		Angle between Direction of Temperature Gradient and Principal Plane Pole - (degrees)		
	No.	Letter	(100)	(010)	(001)		No.	Letter	(100)	(010)	(001)
5-16-52-1	2	D	72.5	57.0	38.5	B272-1	1	A	47.5	42.5	88.5
5-16-52-1	3		64.5	57.0	44.0	B272-1	2	a	33.0	74.0	62.0
5-16-52-1	4		48.5	40.5	87.5	B272-1	3	B	84.5	22.0	69.0
5-16-52-1	1	u	88.5	64.0	26.0	B272-1	4	b	72.0	21.0	81.0
5-16-52-1	5	v	68.0	26.5	77.0	B272-1	5	c	46.0	49.0	72.0
5-16-52-2	2	E	54.5	50.5	59.0	B272-1 tip	1	C	39.0	77.0	54.0
5-16-52-2	4		57.0	53.0	55.0	B272-1 tip	2		40.5	75.5	53.5
5-16-52-2	5		50.0	62.0	53.0	B272-1 tip	9		38.5	76.0	55.5
5-16-52-2	1	w	82.0	61.0	30.0	B272-1 tip	3	d	49.5	83.0	41.5
5-16-52-2	3	x	71.0	28.5	69.5	B272-1 tip	4	e	73.5	49.0	45.5
5-16-52-3	3	F	83.0	18.5	74.0	B272-1 tip	5	f	54.5	59.5	51.0
5-16-52-3	4		80.5	19.0	73.5	B272-1 tip	6	g	64.5	64.0	38.0
5-16-52-3	5		82.0	23.5	68.0	B272-1 tip	7	h	78.5	31.0	62.0
5-16-52-3	1	y	52.5	37.5	88.5	B272-1 tip	8	i	84.0	48.5	42.0
5-16-52-3	2	z	72.0	34.5	61.5	B272-1 tip	10	j	64.0	54.5	47.0
6-12-52-1	3	A	55.0	88.0	35.0	B272-2 tip	1	D	61.5	28.5	88.5
6-12-52-1	6		54.0	83.0	37.0	B272-2 tip	4		60.5	29.5	90.0
6-12-52-1	10		53.0	80.0	39.0	B272-2 tip	2		37.0	54.5	81.5
6-12-52-1	12	a	55.5	75.0	39.0	B272-2 tip	3	m	54.0	40.5	74.5
6-12-52-1	13		50.0	88.0	40.0	B272-2 tip	5	n	36.0	56.0	79.5
6-12-52-1	1		70.5	81.5	22.5	B272-2 tip	6	o	53.5	39.0	75.5
6-12-52-1	2	b	66.0	84.0	24.0	B272-2 tip	7	p	57.5	85.0	33.0
6-12-52-1	7		72.5	86.0	19.5	B272-2 tip	8	q	36.5	55.0	82.0
6-12-52-1	8		70.0	82.0	20.5	B272-2 tip	9	r	26.0	76.0	69.0
6-12-52-1	4	c	65.5	80.5	25.0	B272-4 tip	1	E	66.5	70.5	30.5
6-12-52-1	5	d	67.0	79.5	22.5		4		67.0	69.5	32.0
6-12-52-1	9	e	58.0	87.0	32.0		2	s	82.0	68.0	28.5
6-12-52-1	11	f	71.0	39.5	57.0		3	t	63.5	56.0	46.0
6-12-52-1	14	g	52.5	49.0	62.5		5	u	42.0	70.0	54.5
6-12-52-2	8	B	85.5	66.5	24.5		6	v	53.0	55.0	57.0
6-12-52-2	9		87.5	60.5	29.5		7	w	59.5	71.0	37.0
6-12-52-2	1		77.5	87.5	13.0		8	x	80.0	59.5	32.5
6-12-52-2	2	i	75.0	89.0	15.0		9	y	75.5	47.0	46.0
6-12-52-2	11		66.5	86.0	24.0		10	z	85.0	66.0	24.5
6-12-52-2	3		66.0	88.0	23.5		11	a'	54.5	70.0	43.0
6-12-52-2	4	k	78.0	65.5	13.0		12a	b'	83.5	47.5	43.5
6-12-52-2	5	l	75.0	46.0	47.5		12b	c'	74.0	52.5	42.0
6-12-52-2	6	m	89.0	58.5	32.0		13	d'	60.5	66.0	40.0
6-12-52-2	7	n	67.5	84.5	23.5		14	e'	36.5	72.0	59.0
6-12-52-2	10	o	80.0	73.0	17.5		15	f'	59.0	59.0	46.5
6-12-52-3	5	C	71.0	19.5	84.5		16	g'	90.0	42.0	48.0
6-12-52-3	6	p	76.5	59.0	35.0		17	h'	86.0	79.5	11.0
6-12-52-3	1	g	85.0	54.0	36.5						
6-12-52-3	2	r	76.0	56.0	36.5						
6-12-52-3	4	s	77.0	15.0	83.5						

grain and the rod axis. Figure 10* is a plot of the orientation of the direction in each grain that is parallel to the rod axis (and also to the temperature gradient) with respect to the principal crystal axes.

Figure 10. Grain Orientation Data Plotted in Standard (001) Quadrant Stereographic Projection. (See Table II for correspondence between letter designations and numbers given in Figures 9, 11, 12, 13, 15 and 16.)

a. Specimens 5-16-52-1, -2, and -3

25,690

b. Specimens 6-12-52-1 and 6-12-52-2

25,691

c. Specimen 6-12-52-3

25,692

d. Specimen B 272

22,991

* In these, and in the specimens discussed below, orientations that referred to subgrains of a given grain, as judged by metallographic observations, were averaged and plotted as a single orientation. The largest grain in each specimen is plotted as a capital letter, and the smaller grains as small letters. For correspondence between letter designations and number designations of grain, see Table II.

B. Specimens 6-12-52-1, -2 and -3

This group of samples was prepared from 1/8-inch diameter, 300°C swaged rods. Specimens one-half inch long were cut and formed into "nails" by machining a 60-degree included angle point on one end and peening heads on the other. The specimens were given a preliminary heat treatment of heating into the beta phase and air cooling. Specimen 6-12-52-3 was given an additional heat treatment by heating to 1000°C and water quenching.

For the slow beta-to-alpha transformation treatment, each specimen was sealed in an individual capsule so that the head was supported by a tantalum disc and the machined point was suspended freely. The capsules were affixed to wires, placed into separate furnaces at temperatures within the beta-phase range, and lowered by means of slow-speed motors through temperature gradients into alpha-phase temperature zones. Temperature gradients at the transformation temperature were 96 to 117°C per inch (see Table I).

Composite photomicrographs of the grain structures of these specimens are shown in Figures 11 through 13. The degree of subgraining was somewhat less than in the first group. The orientations of the grains are summarized in Table II and in Figures 10b and 10c in a manner similar to the first group.

C. Specimens B 272-1, -2, -3, -4 and -5

The last group of specimens was prepared from 600°C rolled rods reduced 90% in area to a diameter of approximately 0.300 inch. Two-and-one-half-inch lengths were cut from the rods and machined on one end to form a shoulder of 0.300-inch outside diameter and 0.250-inch inside diameter with a $\frac{1}{2}$ -inch long protruding conical point. The samples were prepared for slow beta-to-alpha transformation as follows: The flat ends of the specimens were inserted into uranium cups, tantalum washers were placed on the machined shoulders, and two-foot long stainless steel tubes were fitted on top of the washers and around the conical points. The assemblies were placed into individual Vycor tubes which were then evacuated to approximately 3×10^{-3} mm Hg pressure and sealed. The uranium cups supported the specimens so that they did not touch the side walls of the Vycor tubes, and the tantalum washers prevented the stainless steel tubes from reacting with the uranium; the stainless steel tubes acted as "heat-sinks" to establish small temperature gradients along the length of the specimens. A macrophotograph of the specimens is shown in Figure 14, and line tracings of the grain structures at several locations are shown in Figures 15 and 16.

The phase-transformation treatment was performed in a lead pot furnace. The capsules were placed inside individual stainless steel tubes

Figure 11. Composite Photo-
micrograph of
Specimen 6-12-52-1

13,628

12.5X

Polarized Light

Figure 12. Composite Photo-
micrograph of
Specimen 6-12-52-2

22,474

12.5X

Polarized Light

Figure 13. Composite Photo-micrograph of Specimen 6-12-52-3

13,627

12.5X

Polarized Light

so that their bottoms were 6 inches below the surface of the 720°C molten lead. The furnace was then cooled to a temperature in the alpha-phase region at a rate of approximately 0.5°C per hour by means of a motor-driven gear train connected to the temperature controller set-point. The temperature gradient at the transformation temperature was 1.3°C per inch (see Table I). The orientation data are summarized in Table II and Figure 10d.

IV. DISCUSSION

The degree of subgraining in the specimens appears to correlate with the magnitude of the temperature gradient along their length. The largest gradient (138°C per inch) was employed in the transformation of specimens 5-16-52-1, -2 and -3, and they showed the greatest amount of subgraining. Specimens 6-12-52-3, -1 and -2 were transformed in measured furnace gradients of 117, 109, and 96°C per inch, respectively; metallographic observations indicated a decreasing degree of subgraining in the same order. In the B 272 specimens which were transformed under a much smaller gradient (1.3°C per inch), the quantity of subgraining was less than that in any of the other specimens.

The magnitude of the temperature gradient also appears to correlate with the sizes and shapes of the alpha grains that were obtained upon slow beta-to-alpha transformation. The samples which were transformed in the largest temperature gradient yielded large grains elongated in the direction of the temperature

gradient. The 6-12-52 samples which were transformed in the next largest temperature gradients also yielded grains elongated in the direction of the temperature gradient, but these were somewhat smaller in size. In the B 272 samples which were transformed in a relatively small temperature gradient the grains were essentially equiaxed.

Figure 14. Macrophotograph of B 272 Specimens. The portions of the specimens that were examined metallographically are outlined in black.

23,598

Bright Field

2X

Figure 15. Line Tracing of Grain Structure on Metallographic Surface of Specimen B 272-1 Bottom. Solid lines represent high angle grain boundaries; dotted lines represent subgrain boundaries. Numbers designate areas whose orientations were determined by Laue back-reflection X-ray photographs.

22,471

17.4X

Figure 16. Line Tracings of Grain Structure Observed on Metallographic Surfaces of Specimens B 272-1, -2 and -4 Tips. Solid lines represent high angle grain boundaries; dotted lines represent subgrain boundaries. Numbers designate areas whose orientations were determined by Laue back-reflection X-ray photographs.

a. Specimen B 272-1 tip

b. Specimen B 272-2 tip

c. Specimen B 272-4 tip

Considering the plots of Figures 10a, 10b and 10c as representing the orientations of grains obtained upon transformation under an imposed appreciable temperature gradient, it is evident that there is a sparsity of points near the (100) pole and a definite tendency for the density of points to increase as the (001) plane pole is approached. In the B272 samples which were transformed in a temperature gradient at least an order of magnitude smaller, the orientations are substantially more random. There appears to be no correlation between grain size and orientation, because the largest grain in each sample assumes the same general distribution as do the smaller grains. The results suggest that an increase in the temperature gradient during slow beta-to-alpha transformation causes a greater concentration of (001) plane poles to lie in the direction of the temperature gradient. It is possible that gradients larger than the ones used in this study would result in an even sharper (001) preferred orientation. The practical implication of this effect is that under conditions of heat treatment that produce temperature gradients one may expect a texture that is not entirely random.

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