

## Scholars' Mine

Masters Theses

Student Theses and Dissertations

1965

# The effect of small alloy additions on the quench induced texture of alpha uranium

Kenneth Ray Riggs

Follow this and additional works at: https://scholarsmine.mst.edu/masters\_theses

Part of the Metallurgy Commons Department:

## **Recommended Citation**

Riggs, Kenneth Ray, "The effect of small alloy additions on the quench induced texture of alpha uranium" (1965). *Masters Theses*. 5230. https://scholarsmine.mst.edu/masters\_theses/5230

This thesis is brought to you by Scholars' Mine, a service of the Missouri S&T Library and Learning Resources. This work is protected by U. S. Copyright Law. Unauthorized use including reproduction for redistribution requires the permission of the copyright holder. For more information, please contact scholarsmine@mst.edu.

## THE EFFECT OF SMALL ALLOY ADDITIONS ON THE QUENCH INDUCED TEXTURE OF ALPHA URANIUM

1731

BY

KENNETH RAY RIGGS

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI AT ROLLA

in partial fulfillment of the requirements for the

Degree of

MASTER OF SCIENCE IN METALLURGICAL ENGLNEERING

Rolla, Missouri

Approved by (advisor) A.5

113008 in a p

#### ABSTRACT

The preferential orientations produced in binary alloys of U-Si and U-Cr on plane sections perpendicular to the direction of heat flow by cooling from beta-phase temperatures were determined. The experimentally determined textures were presented using inverse pole figures.

A definite and reproducible relationship was found to exist between the preferred orientation present at any position and the cooling rate which material at the position had experienced.

It was found that the concentration of the alloying elements within the ranges studied had little detectable effect upon the observed relationship between the cooling rate and the resulting preferential orientation.

It was postulated that the observed relationship between texture and cooling rate in material which had been cooled rapidly resulted from oriented growth of the alpha phase produced by a shear-type beta-to-alpha phase transformation. At lower cooling rates where the transformation became diffusion controlled, the texture was accounted for on the basis of the plastic deformation of the alpha phase due to stresses developed at the beta-to-alpha transformation interface.

ii.

#### ACKNOWLEDGMENTS

This work was done at Mallinckrodt Chemical Works, Uranium Division, Weldon Springs, Missouri under AEC contract number W-14-108-ENG.-8.

The author would like to express his deep appreciation to those individuals at Mallinckrodt Chemical Works, Uranium Division and at the University of Missouri at Rolla who by direct or indirect action have made this work and its culmination possible.

In particular, the author wishes to thank Dr. H. P. Leighly and Dr. N. F. Neumann, who through many stimulating discussions and considerable forbearance have contributed greatly to the quality of this thesis.

## TABLE OF CONTENTS

ABSTRA	cT .	• •	٠	•	•	•	•	•	•	٠	•	•	•	٠	•	•	Page ii
ACKNOW	LEDGM	ENTS	٠	٠	•	٠	٠	٠	•	•	٠	٠	٠	•	•	•	iii
TABLE	OF CO	NTENTS	•	•	•	٠	٠	•	•	•	٠	•	•	٠	٠	•	iv
LIST O	F ILL	USTRAT	TON:	S	٠	•	٠	•	•	٠	•	٠	•	•	•	•	V
LIST O	F TAB	LES	٠	٠	٠	٠	٠	٠	•	•	•	•	•	٠	•	•	vii
1	INTR	ODUCTI	ON	٠	٠	•	٠	٠	•	٠	•	•	٠	٠	•	٠	1
II	REVI	ew of	THE	LI'	<b>r</b> er	ATU	RE	٠	•	٠	٠	•	٠	٠	٠	•	3
III	EXPE	RIMENI	AL	PRO	CED	URE	AN	DR	ESU	LTS	•	•	٠	٠	•	٠	7
	A.	Prefe	rred	d O	rie	nta	tio	n iı	n Al	Lpha	a U	ran	ium	٠	٠	٠	7
	B₊	Retai	ned	Be	ta	Ura	niu	m	٠	•	٠	•	•	•	•	٠	42
IV	DISC	USSION	OF	RE	SUL	TS	٠	٠	•	٠	٠	•	•	•	•	٠	47
v	CONC	LUSION	IS	٠	٠	٠	٠	٠	٠	٠	٠	٠	٠	٠	٠	•	53
	BIBL	IOGRAF	YHY	•	٠	٠	٠	•	•	٠	•	٠	•	•	٠	•	54
	APPE	NDIX	٠	•	٠	٠	٠	٠	٠	•	•	٠	٠	٠	٠	٠	57
	VITA		•	٠	٠	•	•	•	•	•	•	•	٠	•	•	٠	69

## LIST OF ILLUSTRATIONS

Figures				Page
1. Sample shape and dimensions	•	٠	٠	9
2. Argon gas train and sample heating furnace		•	•	10
3. Quenching apparatus	•	٠	٠	12
4. Cooling curves showing one-dimensional cooling of bar	r	٠	٠	13
5. Representative cooling curves for variuos positions in the bar • • • • • • • • • • •	•	•	•	15
6. Cooling rate through 500°C as a function of position in the bar • • • • • • • • • • •	•	•	•	17
7. Positions of plane normals in the standard (001) stereographic pole projection of alpha uranium		•	•	27
8. Average inverse pole figures at 0.010, 0.050, and 0. inches from the quenched surface for samples co 0.08 atomic per cent silicon	100 ont •	) air	ning •	28
9. Average inverse pole figures at 0.150,0.250, and 0.44 inches for 0.08 at. per cent silicon samples	50 •	٠	•	29
10. Average inverse pole figures at 0.010, 0.050, and 0. inches for 0.16 at. per cent silicon samples	•10 •	•	•	30
11. Average inverse pole figures at 0.150, 0.250, and 0. inches for 0.16 at. per cent silicon samples	•45 •	•	•	31
12. Average inverse pole figures at 0.010, 0.050, and 0. inches for 0.51 at. per cent silicon samples	•10 •	•	•	32
13. Average inverse pole figures at 0.150, 0.250, and 0. inches for 0.51 at. per cent silicon samples	•45 •	ю •	•	33
14. Average inverse pole figures at 0.010, 0.050, and 0. inches for 0.04 at. per cent chromium samples	•10	•	•	34
15. Average inverse pole figures at 0.150, 0.250, and 0. inches for 0.04 at. per cent chromium samples	•45 •	60 •	٠	35
16. Average inverse pole figures at 0.010, 0.050, and 0. inches for 0.11 at. per cent chromium samples	•10	•	•	36
17. Average inverse pole figures at 0.150, 0.250, and 0. inches for 0.11 at. per cent chromium samples	•45	•	•	37

18. Average inverse pole figures at 0.010, 0.050, and 0.100 inches for 0.33 at. per cent chromium samples . 38 19. Average inverse pole figures at 0.150, 0.250, and 0.450 inches for 0.33 at. per cent chromium samples . 39 20. Representative variation of grain size with distance from the quenched surface, 0.0 to 0.050 inches from the quenched end μО • • . 21. Representative variation of grain size with distance from the quenched surfsce, 0.100 to 0.500 inches from the quenched end 上1 • 22. Longitudnal section of Jominy bar showing columnar grain structure near the quenched surface 43 23. As-transformed surfaces very near the quenched end in high levels alloy bars 45 . 24. Same as Figure 23. surfaces reground to remove surface relief 46 • •

vi

## LIST OF TABLES

Table		Page
I.	Specimen composition	• 8
II.	Peak positions and scanning ranges	20
III.	Values of A <sub>wi</sub> and I	• 22

#### I INTRODUCTION

The purpose of this investigation was to determine the effects of cooling rate and chemical composition on the preferential crystal orientation produced in alpha uranium by quenching from the beta phase.

During the course of normal reactor operation alpha uranium undergoes dimensional changes. Constant-volume dimensional changes occur in single crystals of alpha uranium when they are irradiated by neutrons<sup>1,2</sup>. Positive growth takes place in the [010] direction and negative growth of an equal magnitude occurs in the [100] direction while the [001] direction remains unaffected. A second type of dimensional change experienced during reactor operation occurs in polycrystalline alpha uranium. This growth or distortion has its origin in the anisotropy of the thermal expansion coefficients of the major crystallographic directions, and is probably due to a thermal ratcheting deformation of individual grains during temperature fluctuations<sup>3</sup>. The dimensional changes exhibited by polycrystalline alpha uranium during reactor operation are therefore dependent upon crystal size and orientation, and from the standpoint of dimensional stability, the most advantageous crystal configuration is one of small, randomly oriented grains.

Forming processes such as extrusion and rolling produce severe preferential orientations or textures in polycrystalline alpha uranium. One heat-treating procedure generally accepted and used for uranium which refines the resulting alpha grain size and essentially

removes textures due to prior working consists of heating uranium containing small amounts of alloying elements into the beta-phase temperature range, holding or soaking for short periods of time at one temperature, and then quenching. After uranium has been heattreated in this manner, however, the resulting alpha structure exhibits a quench-induced preferential orientation, the character of which is dependent upon the severity of the quench.

The successful use of metallic uranium as a nuclear fuel depends to a considerable extent on whether or not it will remain dimensionally stable during reactor operation. Since dimensional instability is due primarily to preferential crystal orientation, information relating preferential orientation to cooling rate and chemical composition is of considerable value.

#### II REVIEW OF THE LITERATURE

The determination and representation of preferential orientations in polycrystalline metals has been the object of a considerable number of investigations in the last 25 years. Several reviews of the various techniques which have evolved are available 4,5,6.

The use of the ratios of measured intensities and calculated random intensities as an internal standard in determining the relative preferential orientation of crystallographic planes was first suggested by Harris<sup>7</sup>. The expression derived by Harris for the texture coefficients of individual crystallographic planes was corrected by Morris<sup>8</sup>. Morris later introduced the use of "area weighting factors" in the mathematical development<sup>9</sup>. The area weighting factors were introduced in order to reduce the effects of a non-uniform distribution of sampling points upon the resulting calculated orientations and to insure that all possible crystal orientations were accounted for in the formalism. Several discussions of the assumptions involved in this particular method of representing preferential orientations and the inherent limitations of the method have been published<sup>10,11</sup>. The "area weight" method of calculating texture coefficients as developed by Harris and Morris was used to represent the preferential orientations measured in this study. A fairly complete description of this method is given on pages 19 through 25 below.

Holden<sup>12</sup> has compiled an excellent review of work done on the physical and chemical properties of uranium prior to 1957. The

discussion below is intended to include information concerning only those properties of uranium which have a direct effect upon the orientation present in alpha uranium after cooling from the beta phase.

The equilibrium beta-to-alpha phase transformation in pure uranium has been reported to occur at  $663^{\circ}C^{13}$ . The effects of small alloy additions on the equilibrium beta-to-alpha phase transformation can be seen in the equilibrium phase diagrams for binary alloys of U-Si and U-Cr containing small concentrations of these alloying elements<sup>14,15</sup>. An examination of the equilibrium phase diagrams reveals that at  $740^{\circ}C$ all the alloys investigated were within the one phase region of solid solubility.

Several investigations have been made which indicate that heating uranium into the beta phase temperature range will essentially remove textures found in the alpha phase<sup>16,17</sup>. Since heating uranium into the beta phase removes textures originally found in the alpha phase, any systematic and reproducible texture found in the alpha phase after quenching must have been introduced by the quench.

The chemical and physical properties of the beta and alpha phases and the sudden change in these properties at the beta-to-alpha phase transformation must surely have a great deal to do with the resulting texture seen in the alpha phase. Beta uranium has a complex tetragonal crystal structure containing 32 atoms per unit cell<sup>18</sup> and therefore exhibits few deformational modes<sup>19</sup>. Beta uranium has been shown to be harder <sup>20</sup> and to have a higher yield strength<sup>21</sup> than the alpha phase at the phase transformation temperature. The crystal structure of alpha uranium has been determined to be orthorhombic<sup>22</sup>. This phase exhibits numerous deformational modes - both by slip and by twinning<sup>23,24</sup>.

Calnan and Clews<sup>25</sup>, by considering the known deformation mechanisms of alpha uranium, have predicted the orientations which should result from deformation involving both slip and twinning when tensile stresses are applied to randomly oriented samples of polycrystalline alpha uranium. Since deformation of alpha uranium proceeds mainly by slip above  $450^{\circ}$ C with twinning being favored as the deformational mode below this temperature<sup>26</sup>, the preferential orientations which would result from tensile stresses in the alpha phase, according to Calnan and Clews, would consist of the plane normals of planes close to the (110) being oriented parallel to the direction of applied stress for tensile stress applications at temperatures above  $450^{\circ}$ C. For tensile stress applications at temperatures below  $450^{\circ}$ C, the plane normals of planes close to the (010) plane should be oriented parallel to the direction of stress application.

The kinetics of the beta-to-alpha phase transformation in binary alloys of U-Cr and U-Si have been studied<sup>27,28</sup>. It was found that alloys containing very small concentrations of either alloying element transformed by a shear-type process at low temperatures and by a diffusion controlled process at higher temperatures.

The exact nature of the beta-to-alpha phase transformation in pure uranium at various temperatures has been the subject of considerable debate. Burke and Dixon<sup>29</sup> have compiled a fairly complete review of

the literature on this subject and have concluded that the beta-toalpha transformation in pure uranium proceeds by a shear-type process at high cooling rates (low transformation temperatures) and that at lower cooling rates (higher transformation temperatures) the reaction becomes diffusion controlled.

Butcher, et. al.<sup>30</sup>, in quenching studies of uranium containing low levels of impurities have observed columnar grains, elongated in the direction of heat flow in areas of high cooling rate. The investigators postulated that oriented growth of the martensitically transformed alpha occured in these regions.

Russell<sup>31</sup> in an extensive investigation of the properties of the alpha phase produced by quenching from beta-phase temperatures found columnar grains which were elongated in the direction of heat removal in areas near the quenched surface of rods and tubes of uranium containing low levels of impurities. In studying the preferential orientations which resulted from quenching, Russell also observed that in areas of high cooling rate, (200) planes were preferentially oriented perpendicular to the direction of heat flow or parallel to the quenched surface. As the cooling rate decreased at positions farther from the quenched surface, the texture decayed by movement of the area of primary excess texture along the [010] zone to the (002) plane and then along the [100] zone toward the (020) plane. Kornfeld<sup>32</sup> in quenching studies on Jominy bars of uranium containing low levels of impurities was the first to report this relationship between texture and cooling rate.

#### III EXPERIMENTAL PROCEDURE AND RESULTS

## A. PREFERRED ORIENTATION IN ALPHA URANIUM.

Binary alloys of U-Cr and U-Si (Table I) were investigated. Preferential crystal orientations were produced in cylindrical samples of these alloy systems by a Jominy end-quench from the beta phase. The resulting crystal orientations in cross-sectional areas parallel to the quenched surface were determined using x-ray diffraction techniques. Duplicate samples of each composition were examined to determine the reproducibility of the data.

In order to simplify the problem of correlating the observed structure and measured cooling rates, it was desirable to use a method of quenching that gave the simplest relationship between direction of heat removal and sample geometry. It was also desirable that the quenching procedure produce a wide variation of cooling rates within the same sample since this condition would facilitate a study of the effects of different cooling rates upon the structure while greatly reducing the number of samples to be heat-treated.

The standard Jominy hardenability test quenching procedure, a method of quenching which produced both of the desired cooling conditions, was used. The samples were cylinders (Figure 1) which were machined from  $1-1/l_1$ -inch-diameter rods that had been formed by extrusion at gamma-phase temperatures.

Each sample was heated by suspending it in a vertical tube furnace under an atmosphere of preheated and purified argon gas (Figure 2). The cylinder was positioned so that the bottom 1/2 inch

## TABLE I

## SPECIMEN COMPOSITION

Specimen	No.		Analysis (p	om by wt.)	
	*****	Si	C <b>r</b>		Fe
1	90	- 0.08 at.%	16	$ND^{a}$	140
2	190	- 0.16 at.%	15	ND	130
3	600	- 0.51 at.%	13	ND	70
24		48	97 - 0.04 at.%	31	64
5		29	230 - 0.11 at.%	ND	67
6		28	715 - 0.33 at.%	ND	59

a ND = not detectable

analyses furnished by the spectrochemical section, Mallinckrodt Chemical Works, Uranium Division





Figure 1 Sample shape and dimensions



Figure 2 Argon gas train and sample heating furnace

rested at the center of the furnace. The sample was heated at a rate of approximately  $15^{\circ}$ C per minute to  $740^{\circ}$ C and held at this temperature for an hour. The variation in temperature around the control point at the center of the furnace was  $\pm 5^{\circ}$ C.

While the sample was being heated to temperature, the shutter in the quenching apparatus (Figure 3) was moved from the position above the nozzle and the pumped stream of water was adjusted to an unrestrained height of 2-1/2 inches. The shutter was then placed in the path of the stream and the quench was initiated by manually placing the sample (transfer times were approximately  $l_1$  seconds) in position in the quenching apparatus and flipping the shutter aside. The sample was left in position in the quenching apparatus for a period of one hour to insure that the whole bar was cooled to the temperature of the water. The water in the holding tank of the quenching apparatus remained at room temperature.

The end-quenching procedure described above produced a condition in the cylinder near the quenched surface that closely approximated one-dimensional cooling. Cooling curves were obtained that showed that one-dimensional cooling was realized for the portion of the bar within 1/2 inch of the quenched surface (Figure 4).

The variation of cooling rate with distance from the quenched surface was determined by placing unsheathed Chromel-Alumel thermocouples at the mid-radius of the cylinder and at varying distances from the quenched surface. The change of temperature with cooling time was measured with potentiometric recorders.



top view: no sample in holder





Figure 3 Quenching apparatus, schematic



The thermocouples were constructed of 28 gauge wire and 1/16inch-diameter ceramic electrical insulators. The thermocouples were force-fitted in 1/16-inch-diameter holes that were drilled radially to a depth of 1/4 inch.

The positions chosen for the measurement of cooling rates were 0.05, 0.10, 0.25, and 0.50 inches from the quenched surface. The recorders used for determinations at the 0.05- and 0.10-inch positions had chart speeds of one inch per second and were capable of measuring temperature changes in the range 0 to  $3000^{\circ}$ C per second. The recorders used for the 0.25- and 0.50-inch positions had chart speeds of 8 inches per minute and were capable of measuring temperature changes in the range 0 to  $650^{\circ}$ C per second.

Just prior to the start of the quench, a check was made to insure that good thermal contact existed between the thermocouple bead and the metal sample. This check consisted of measuring the electrical resistance between the copper sample hanger and each of the thermocouple lead wires at positions just outside the sample heating furnace. Each thermocouple in each quench was found to have excellent electrical contact - and therefore good thermal contact - with the metal sample.

After several sets of cooling curves had been determined, it was noticed that the alloy content within the ranges of interest had little detectable effect upon the quench rate. This made it possible to reduce the number of temperature versus time determinations and to draw one set of cooling curves which were representative of all the alloy systems. The resulting set of cooling curves (Figure 5)



represent an average of six such determinations.

Since the primary purpose of this portion of the work was to correlate cooling rate and the resulting crystal orientation, it was necessary to choose a particular temperature and use the cooling rate through this temperature as a reference. It was intended that the cooling rate just prior to the beginning of the beta-to-alpha phase transformation be used for reference. An examination of Figure 5 shows, however, that the change in slope of the cooling curve indicative of the beta-to-alpha phase transformation was not detected in this study. Since the transformation was not detectable, a temperature of  $500^{\circ}$ C was arbitrarily chosen and the cooling rate through this temperature was used as a reference. Figure 6 shows the variation of cooling rate through  $500^{\circ}$ C with distance from the quenched surface.

After quenching, the variation of crystal orientation with distance from the quenched surface in each cylinder was determined. The surfaces examined were cross-sectional areas which were parallel to the quenched surface and therefore perpendicular to the direction of heat flow. Only material within 1/2 inch of the quenched surface was examined since that was the region of greatest interest and because the heat flow in the bar at positions farther than 1/2 inch from the quenched surface ceased to be one-dimensional.

Axial lengths of 5/8 inch were cut from the bottoms of the cylinders. In reaching cross-sections of interest in these samples, metal was removed from the transverse surface of the cylinders by simultaneously grinding a number of samples in a Beuhler Automet.



The bulk of the metal was removed by grinding on 180 grit silicon carbide grinding paper. As a cross-section of interest was approached, however, the sample surfaces were ground on 320 and then on 600 grit paper in order to minimize the amount of disturbed metal on the surface produced by grinding. After repeated grinding of the same sample, it was found that the resulting surfaces remained parallel to within ± 0.003 inches at the cylinder wall.

After grinding on the 600 grit paper, the resulting surface was electropolished in a solution of chromic and acetic acids (118 grams  $CrO_3$ , 100 ml.  $H_2O$ , 400 ml. glacial acetic acid) with a sample-to-cathode separation of approximately 2 inches. The surface was polished for 2 minutes at approximately 40 volts.

The electropolished sample was placed in the 1-inch-diameter rotating specimen holder of a Philips Electronics diffractometer and the sample surface was checked to insure that it was free of disturbed metal.

The check of the sample surface consisted of scanning through an angular range of 20 in which the diffracted peak from the (110) plane of the alpha phase was known to fall. When fairly sharp resolution of the diffracted Cu K $\alpha_1$  and K $\alpha_2$  lines was observed, it was assumed that the surface was free of disturbed metal. If resolution of these peaks was not obtained after the initial electropolishing, the sample was re-electropolished for periods of 30 seconds until resolution was obtained.

Nickel filtered Cu Ko x-radiation was used in the examination.

The operating voltage was 40 kilovolts and the operating current was 20 milliamps. A scanning speed of  $1^{\circ}$  20 per minute was maintained during examination and a  $1^{\circ}$  divergence slit was used in the diffractometer. The specimen holder was positioned so that the center of the x-ray beam fell on the mid-radius of the electropolished surface. During the course of the examination, the cylinder was rotated about its axis at a rate of approximately 200 revolutions per minute. Since the height of the x-ray beam striking the specimen surface was about 3/8 inch, the positioning of the sample with respect to the beam and the rotation permitted analysis of approximately 75% of the sample surface.

The determination of the crystal orientations in the work-free electropolished surface was accomplished using the same operating conditions for the sample holder and diffractometer as listed above. The integrated intensities diffracted from 18 crystallographic planes in the sample surface were measured. The integrated intensity from a particular plane was measured by scanning over a range of 20 in which the diffracted peak from the plane was known to fall (Table II). The integrated intensity was the accumulated intensity measured during the scan minus the background intensity.

Measurements of background intensity were made at  $33^{\circ}$ ,  $56^{\circ}$ ,  $96^{\circ}$ , and  $123.5^{\circ}$  20. The background intensity measured at  $33^{\circ}$  was subtracted from the total intensities of the (020) thru (111) peaks. The background intensity at  $56^{\circ}$  was subtracted from the peaks (112) thru (132). The background intensity measured at  $96^{\circ}$  was used for the remainder of the peaks with the exception of the (312). The

### TABLE 11

PEAK	POSITIONS	AND	SCANNING	RANGES

(hkl)	Peak Position - 20	Scanning Range (°20)
020	30.46	30.0 - 31.0
110	34•96	34.2 - 35.2
021	35.55	35.2 - 35.95
002	36•26	35.95 - 36.95
111	39•54	39.0 - 40.0
112	51.24	50 <b>.7</b> 5 - 51.75
130	57.08	56.6 - 57.6
131	60.32	59.8 - 60.8
023	64.76	64.0 - 65.0
200	65.40	65.0 - 66.0
113	67.42	67.0 - 68.0
132	69.52	69.0 - 70.0
133	83.80	83.4 - 84.4
114	87.42	87.0 - 88.0
150	90.49	90.0 - 91.0
223	<b>99.0</b> 5	98.65 - 99.65
152	101.66	101.20 - 102.20
312	122.78	122.00 - 123.50

The O2O, 002, and 20O planes are Bragg planes and the intensities from these are second order reflections from the (010), (001), and (100) Miller planes. The remainder of the planes examined are Miller planes.

background at 123.5° was used for the (312) peak only.

The measured integrated intensities from the crystallographic planes were used to calculate the "texture coefficients" of the crystallographic planes in the surface examined:

$$\alpha_{i} = \frac{I_{i}/I_{i}}{\sum_{i=1}^{k} A_{wi} I_{i}/I_{i}}, \qquad 1)$$

where  $\alpha_i$  = the texture coefficient of a particular plane,  $I_i$  = the measured integrated intensity,  $I'_i$  = the calculated intensity from a randomly oriented plane, and  $A_{wi}$  = the "area weighting factor" of the plane. The values of  $A_{wi}$  and  $I'_i$  used in this investigation are given in Table III. A partial derivation and discussion of the expression for the texture coefficient follows below.

The expression for the diffracted intensity from the surface of a thick, randomly oriented powder specimen, as determined using a diffractometer is:

$$I' = \left(\frac{I_{0}e^{4}}{m^{2}c^{4}}\right)\left(\frac{\lambda^{3}A}{v^{2}}\right)\left(\frac{1}{16\pi r \sin^{2}2\theta \cos\theta}\right)\left(\frac{1 + \cos^{2}2\theta}{2}\right)\left(F^{2}p\right)\frac{e^{-2M}}{2(\mu + \mu_{s})}$$

where I' = 'integrated intensity' or total energy entering the counter,  $I'_0$  = intensity of the incident beam, e and m = charge and mass of the electron, c = velocity of light,  $\lambda$  = wavelength of incident radiation, r = radius of the diffractometer circle, A = cross-sectional area of the incident beam, v = volume of a unit cell, l = length of the diffraction cone analyzed (= length of the receiving slit), (1/16 r sin<sup>2</sup>20 cos0) = Lorentz factor, 0 = Bragg angle, F = structure factor, (1 + cos<sup>2</sup>20/2) = polarization factor, p = multiplicity,

	TABLE III	
	VALUES OF A and	(10) 1 I <u>1</u>
(hkl)	Awi	I <mark>i</mark>
020	<b>•031</b> 8	6.34
110	•0634	72•7
021	•0608	100.0
002	•030l4	51.4
111	•0738	58.3
112	•0522	48.3
130	•0357	3.37
131	•0684	<u>40•0</u>
<b>0</b> 2 <b>3</b>	•0659	16.8
200	•0234	8.82
113	•0477	11.6
132	•0529	3.65
133	•0795	15.3
114	•0749	10.2
150	•0372	7.43
223	•0487	12.2
<b>1</b> 52	•0689	12.8
312	•0845	8,85

 $e^{-2M}$  = temperature factor,  $\mu$  = linear absorption coefficient,  $\mu_s$  = correction factor for secondary extinction.

For the size sample which is conveniently analyzed using the diffractometer, the assumption that a random orientation exists presupposes that the sample is composed of small crystals of essentially uniform size and that an equal number of crystals are in every possible orientation with respect to any reference direction.

From the derivation of the Lorentz factor<sup>33</sup>, it can be seen that for a randomly oriented sample, the volume of material oriented such that the diffracted beam from a particular (hkl) plane will fall within a length, 1, of the diffraction cone is given by:

$$\rho' = 1/8\pi r \sin\theta_{\bullet}$$
 3)

For a particular set of physical conditions, <u>i.e.</u> temperature, incident intensity, and goniometer adjustment, the expression for the integrated intensity from the surface of a randomly oriented polycrystalline sample becomes:

$$I' = K' \rho',$$
where  $K' = (I_o^{i} e^{i/4} / m^2 c^{i/4}) (\lambda^3 A / v^2) (1 + \cos^2 2\theta / 2\sin 2\theta) (F^2 p') e^{-2M} / 2(\mu + \mu_s').$ 

We can now write a new function:

$$I/I^{i} = (K/K^{i})(\rho/\rho^{i})$$

$$= c(\rho/\rho^{i})$$

$$= c \alpha,$$
5)

where  $C = (I_0/I_0)(p/p')(\mathcal{M} + \mathcal{M}_S'/\mathcal{M} + \mathcal{M}_S)$ . The unprimed terms represent a particular crystallographic plane in a preferentially oriented sample and the primed terms represent the same plane in a randomly oriented sample.

Harris<sup>7</sup> has suggested that the value of C can be approximated by assuming that the mean value of  $\alpha$  (for a number of different crystallographic planes) is 1:

$$\frac{1}{n} \sum_{i=1}^{m} \mathbf{I}_{i} / \mathbf{I}_{i}^{i} = \frac{1}{n} \sum_{i=1}^{m} C_{i} < i$$

$$= C \frac{1}{n} \sum_{i=1}^{m} i$$

$$= C_{i}$$

$$= C_{i}$$

$$= C_{i}$$

so that

$$\alpha_{i} = (I_{i}/I_{i}^{\dagger})/\frac{1}{n} \sum_{i=1}^{n} (I_{i}/I_{i}^{\dagger}).$$
 7)

Correcting for the non-uniform distribution of sampling points, after Morris<sup>9</sup>, gives  $\alpha'_{i} = (I_{i}/I_{i}^{i}) / \sum_{i=1}^{n} A_{wi} I_{i}/I_{i}^{i}$ . 8)

In the course of the above derivation, it was assumed that the value of C is independent of any particular (hkl) plane. This assumption implies that the quantity  $(p/p')(\mu + \mu'_s/\mu + \mu_s)$  is independent of the degree of texture of the polycrystalline aggregate. This assumption introduces only small errors when the metal examined has low crystal symmetry and a large linear absorption coefficient.

A knowledge of the value of  $(\rho / \rho')$  or  $\alpha_i$  is very useful since the magnitude of this term indicates the degree to which the particular crystallographic plane is aligned parallel to the specimen surface examined. A value of  $\alpha_i < 1$  indicates that the crystallographic plane is preferentially deficient and a value of  $\measuredangle_i$ >1 indicates that the plane is preferentially in excess in the surface examined. A value of  $\measuredangle_i = 1$  indicates that the particular crystallographic plane is randomly oriented in the surface examined.

18 texture coefficients - one for each crystallographic plane examined - were calculated from the intensity measurements taken from each surface. Since duplicate samples of each alloy were studied, it was possible to determine the average values of the texture coefficients at selected positions in the bars. Due to the difficulty unvolved in grinding to preselected positions in the bars, the duplicate samples were not examined at identical distances from the quenched surface. The average values of the texture coefficients for duplicate bars of identical composition were therefore determined graphically. The average values of the texture coefficient of a particular plane were determined by plotting the variation of the texture coefficient with distance from the quenched surface. Values for the two bars of identical chemistry were plotted together and the average values at selected positions on the plot were determined.

One very useful method of presenting the orientation as given by the texture coefficients of the planes examined consists of plotting the values of the texture coefficients on a standard stereographic pole projection. When the texture coefficient of each plane is plotted at the position of the plane normal in a standard stereographic pole projection, a quantitative "picture" of the orientation results. A plot of this type has been called an "inverse pole figure". A standard (001) pole projection was used to present the average orientations found in the alloys investigated (Figures 7 thru 19). Figure 7 shows the positions of the plane normals of the planes examined on the standard (001) pole projection. Figures 8 thru 19 show the average values of the texture coefficients plotted at the appropriate pole positions. The values of  $\alpha_i$  from the individual surfaces examined are tabulated in the Appendix.

Each surface in which the crystal orientations were determined was also subjected to metallographic examination using polarized light. Photomicrographs of the as-electropolished surfaces were taken (Figures 20 and 21).



Figure 7 Positions of the plane normals in the standard (001) stereographic pole projection of alpha uranium


























0.013 inches from quenched surface cooling rate through 500°C = 400°C/sec. 50 x, polarized light



0.048 inches from quenched surface cooling rate through 500°C = 170°C/sec. 50X, polarized light

Representative variation of grain size with distance from the quenched surface, 0 to 0.050 inches from the quenched end



0.109 inches from quenched surface cooling rate through 500°C = 85°C/sec. 50X, polarized light



0.273 inches from quenched surface cooling rate through 500°C = 25°C/sec. 50X, polarized light

Representative variation of grain size with distance from the quenched surface, 0.100 to 0.500 inches from the quenched end

## B. RETAINED BETA URANIUM.

Cylindrical samples of uranium containing 0.08 atomic per cent silicon were heat-treated and quenched in the manner described above. The cylinders were then cut along their longitudinal axes and the crystal structures of these longitudinal sections were examined (Figure 22).

Samples containing 0.51 atomic per cent silicon and others containing 0.33 atomic per cent chromium were studied in an attempt to determine the nature of the beta-to-alpha phase transformation in regions of high cooling rate - close to the quenched surface. These samples were heated into the beta phase to 740°C, held at this temperature for an hour, and then quenched. The duration of the end-quench in this investigation was 5 minutes. After end-quenching for 5 minutes the bar was cooled quickly by submerging it in the water in the holding tank. An axial length of approximately 5/8 inch was cut from the bottom of each cylinder. The quenched surface was ground lightly on 600 grit grinding paper and electropolished. The electropolished surface was examined by means of x-ray diffraction to determine if the beta phase was retained by the high cooling rates at the quenched surface. The elapsed time between the completion of quenching and the start of x-ray examination was approximately 30 minutes. Repetitive scans were made over the angular range 37°to  $40^{\circ}$  20. The (411) and (212) peaks from the beta phase and the (111) peak from the alpha phase all fall in this angular range.

Retained beta phase was found to exist at the quenched surface in both the alloys investigated. The retained beta phase transformed



0 to  $\sim 0.090$  inches from quenched surface 50X, polarized light

Longitudinal section of Jominy bar showing columnar grain structure near the quenched surface quite slowly at ambient temperature and from the roughening of the sample surfaces which occurred during transformation, it was ascertained that the transformation proceeded by a shear-type mechanism. After the apparent completion of transformation, as shown by the disappearance of the (411) and (212) beta peaks, photomicrographs were taken of the as-transformed surfaces (Figure 23). These surfaces were then ground lightly on 600 grit paper to remove the surface relief due to transformation and then examined again (Figure 24).



0.33 atomic per cent chromium 50X, polarized light



0.51 atomic per cent silicon 50X, polarized light

As-transformed surfaces very near the quenched end in high level alloy bars



0.33 atomic per cent chromium 50X , polarized light



0.51 atomic per cent silicon 50X, polarized light

Same as Figure 23, surfaces reground to remove surface relief

## IV DISCUSSION OF RESULTS

The areas of excess pole density on the inverse pole figures are those areas in which the texture coefficients have values greater The areas of excess pole density therefore represent than unity. those crystallographic planes which are preferentially oriented parallel to the surface examined. As can be seen by referring to the inverse pole figures presented above, there is a definite relationship between the cooling rate and the position of the area of excess pole density. The relationship between the cooling rate and the preferential orientation - a relationship which was common to all the alloys examined - can be described as follows: In regions of high cooling rate near the quenched end of the bar, (200) planes are oriented parallel to the quenched surface. The measured texture coefficients of the (200) plane on cross sectional areas close to the quenched surface are therefore greater than 1. Examination of the inverse pole figures for the cross section at 0.010 inches from the quenched surface shows that there are approximately 3 times as many grains oriented with the (200) plane parallel to the surface than there would be if the material at the surface were randomly oriented. As the cooling rate decreases at positions farther from the quenched surface, the area of primary excess pole density on the inverse pole figures moves from the (200) pole position along the [010] zone toward the (002) pole position. The texture coefficient of the (002) plane reaches a maximum value at approximately 0,150 inches from the quenched surface where the cooling rate through 500°C has dropped to about 50°C per second. In this region of the bar, the (002) planes

are preferentially oriented parallel to the quenched surface - or perpendicular to the direction of heat flow. With increasing distance from the quenched surface, the area of primary excess texture moves from the vicinity of the (002) pole position along the [100] zone toward the (020) pole position. In these regions of the bars where the cooling rate is relatively low, the area of primary excess texture becomes increasingly larger and at the same time the texture coefficients of those planes represented by the area of primary excess texture decrease until at approximately 0.450 inches from the quenched surface the texture in the bars degenerates to a quasi-random state.

The most likely source of the textures developed in alpha uranium during rapid cooling from the beta phase is the stress developed at the beta-to-alpha phase transformation interface. The volume change on going from the beta to the alpha phase at the equilibrium temperature is approximately 2%. When the beta-to-alpha transformation occurs at low temperatures where the thermal mobility of the atoms is reduced, the stresses developed at the transformation interface will be quite large. The stresses developed at the transformation interface will be highest in regions of high cooling rate and will decrease in magnitude as the cooling rate decreases.

If the phase interface is assumed to remain essentially planar and parallel to the quenched surface, the stresses at the interface will be radial. Under these conditions the beta phase at the interface will be in radial compression and the alpha phase will be in radial tension. The transformation interface will not, of course, remain completely planar; however, since a fairly steep temperature

gradient exists along the axis of the bar with no gradient being present in a radial direction, the transformation interface will always tend to move parallel to the axis of the cylinder and will also tend to remain planar. It is therefore proposed that the stress condition at the beta-to-alpha phase interface consists mainly of radial compression in the beta phase and radial tension in the alpha phase.

From the quenching studies conducted on the high-level alloy bars, it is evident that the transformation in these alloys in regions of high cooling rate proceeds by a martensitic or shear-type process. It is reasonable to expect that the nature of the phase transformation will have a profound effect upon the preferential orientation produced in alpha uranium by quenching from the beta phase, and since a marked similarity exists between the preferential orientations found in all the alloys, it is concluded that the phase transformation in areas of high cooling rate in all the alloys investigated proceeds by a shear-type process. The grain structure of the metal in the regions of high cooling rate consists of crystals which have small transverse diameters but which are markedly elongated in the direction of heat flow. The grains extend from the quenched surface back to about 0.080 inches from the quenched surface. Since the material in this region has been shown to transform by a shear-type process, the only possible explanation of the elongated or columnar grains is that oriented or preferential growth of the martensitically transformed alpha occurs. As can be seen from the average inverse pole figures presented earlier, the preferred direction of martensitic growth lies near the [100] direction in alpha uranium. Since the beta

structure produced by the alpha-to-beta phase transformation is essentially random  $^{16,17}$ , the idea that the textures found in the samples within 0.080 inches of the quenched surface resulted from an oriented <u>nucleation</u> of the alpha phase on a preferred habit plane of the beta phase must be discounted.

From a consideration of the change of grain shape and orientation which occurs at approximately 0.080 inches from the quenched surface, it is concluded that the beta-to-alpha phase transformation in this region changes from a shear-type process to one that is diffusion controlled. Added weight is given to this conclusion by the observation that the texture in the samples at distances greater than approximately 0.080 inches from the quenched surface can be accounted for in terms of the plastic deformation of the alpha phase resulting from the stress produced at the beta-to-alpha transformation interface. Since the beta phase is both harder and stronger than the alpha phase at the transformation temperature, it follows that plastic deformation occuring near the transformation interface will take place primarily in the alpha phase.

Calnan and Clews<sup>25</sup>, from a consideration of the known deformation mechanisms of alpha uranium, have concluded that when twinning predominates as the deformational mechanism, which does occur at temperatures below 450°C, the (010) planes and planes lying at small angular distances from the (010) plane in the unit cell will be preferentially aligned perpendicular to the direction of applied stress. At temperatures above 450°C, where slip predominates as the deformational mode, planes in the neighborhood of the (110) plane will be preferentially aligned perpendicular to the direction of applied stress.

Since the direction of the tensile stress in the alpha phase at the transformation interface is radial, the following orientations should exist in those portions of the bars where the orientation results from plastic deformation of the alpha phase:

- In regions of high cooling rate where the transformation temperature is low, planes in the neighborhood of the (020) plane should be oriented perpendicular to the direction of heat flow. The texture coefficients of these planes in the surfaces examined should therefore be less than unity.
- 2. In regions of intermediate cooling rates both twinning and slip should occur near the transformation interface. In these portions of the bars, the texture coefficients of planes near the (020) plane should remain low and the texture coefficients of planes near the (200) plane should become smaller. Since both the (020) planes and (200) planes should show a tendency to align themselves perpendicular to the surfaces examined, the texture coefficient of the (002) planes in this region should become larger than unity.
- 3. In regions of low cooling rates planes near the (200) plane should tend to be oriented perpendicular to the surfaces examined. The texture coefficients of planes near the (020) plane should therefore become larger and the texture coefficients of planes near the (002) should become smaller.

The stress level drops continuously with increased distance from

the quenched surface so that the change in orientation in going from region 1. to region 3. is continuous. The magnitude of the texture developed should decrease with increasing distance from the quenched surface until a quasi-random structure results.

Comparison of the predicted textures discussed above and the experimentally determined textures at distances greater than 0.080 inches from the quenched surface, as given by the inverse pole figures, shows that very good agreement exists between theory and experiment.

The concentration of the alloying elements, silicon and chromium, had little detectable effect upon the observed relationship between cooling rate and preferential crystal orientation. The concentrations of the alloying elements were low and at  $7{10}^{\circ}$ C - the soaking temperature employed in this study - all of the alloys investigated were in a single phase region of solid solubility. Other investigators have shown<sup>27,28</sup> that the relationships between the transformation kinetics and transformation temperature should be quite similar in the six alloys investigated in this study. It is not surprising, then, that there was little detectable effect of alloy concentration upon the observed preferential orientations.

#### V CONCLUSIONS

Alloying additions of silicon and chromium within the concentration ranges studied in this investigation have little detectable effect upon the preferential crystal orientation of alpha uranium produced by quenching from the beta phase.

A definite and reproducible relationship between cooling rate and observed preferential orientation was found to exist in all the alloys examined. In regions of high cooling rate, the area of primary excess texture on the inverse pole figures was centered around the (200) pole position. As the cooling rate decreased, the area of primary excess texture moved along the [010] zone to the (002) pole position and then along the [100] zone toward the (020) pole position. For very small cooling rates, the texture degenerated to a quasi-random state.

In those regions of the bars where the cooling rate through 500°C exceeded 110°C per second, an oriented growth of the martensitically transformed alpha was found to occur. The direction of preferred growth of the martensitically transformed alpha was close to the [100] direction in alpha uranium.

At cooling rates less than approximately  $100^{\circ}$ C per second the transformation appeared to occur primarily by a diffusion controlled process. The preferential orientations found in those regions where the cooling rate through  $500^{\circ}$ C was less than  $100^{\circ}$ C per second were concluded to result from the plastic deformation of the alpha phase due to the stresses developed at the transformation interface.

### BIBLIOGRAPHY

- S. H. PAINE and J. H. KITTEL (1958) "Preliminary Analysis of Fission-Induced Dimensional Changes in Single Crystals of Uranium" AEC Report ANL-5676.
- 2. A. M. TURKALO (1954) "The Dimensional Stability of Uranium Single Crystals and Polycrystals During Irradiation and Thermal Cycling" AEC Report KAPL-1044.
- 3. J. E. BURKE and A. M. TURKALO (1952) "Deformation of Zinc Bicrystals by Thermal Ratcheting" Trans. Am. Inst. Mining Met. Engrs. Vol. 194, p. 651.
- 4. B. D. CULLITY (1956) <u>Elements of X-Ray</u> <u>Diffraction</u>. Addison-Wesley, Reading, Mass., p. 272-295.
- 5. C. S. BARRETT (1952) <u>Structure of Metals</u>. McGraw-Hill, New York, p. 170-195.
- 6. A. TAYLOR (1961) X-Ray Metallography. Wiley, New York, p. 561-655.
- 7. G. B. HARRIS (1952) "Quantitative Measurement of Preferred Orientation in Rolled Uranium Bars" Phil. Mag. Vol. 43, p. 113-123.
- 8. P. R. MORRIS (1953) "An Internal Standard For the Determination of the Proportionality Constant in Preferred Orientation Studies" AEC Report FMPC-310.
- P. R. MORRIS (1959) "Reducing Effects of Nonuniform Pole Distribution in Inverse Pole Figure Studies" Jour. of Applied Phy. Vol. 30, p. 595-596.
- 10. R. N. THUDIUM and P. R. MORRIS (1961) "Brief Description of the "Area-Weight" Treatment For the 18-Plane Set" AEC Report ANL-6359, p. 55-57.
- 11. P. R. MORRIS (1960) "Editors Introduction" AEC Report NLCO-804 p. 7-8
- 12. A. N. HOLDEN (1958) Physical Metallurgy of Uranium. Addison-Wesley, Reading, Mass.
- 13. J. E. BAUMRUCKER and H. H. CHISWIK (1953) "Thermal Analysis of High Purity Uranium" AEC Report ANI-5234, p. 30-33.
- 14. J. A. STRAATMANN and N. F. NEUMANN (1964) "Equilibrium Structures in the High Uranium-Silicon Alloy System" AEC Report MCW-1486.

- 15. T. A. BADAEVA and R. I. KUZNETSOVA (1963) "Constitutional Diagrams of Uranium-Chromium Alloys" in <u>Structure</u> and <u>Properties</u> of the Uranium, <u>Thorium</u>, and <u>Zirconium</u> <u>Alloys</u>. Moscow, Gosatomizdat, p. 87-91.
- 16. W. R. McDONELL (1962) "Kinetics of Structural Changes During Beta Transformation of Uranium" Nuc. Science and Engineering Vol. 12, p. 325-336.
- 17. I. F. BARWOOD and B. R. BUTCHER (1963) "The α-β Phase Transformation in Uranium" Jour. of Nuc. Materials Vol. 8, p. 232-240.
- C. W. TUCKER and P. SENIO (1953) "An Improved Determination of the Crystal Structure of Beta-Uranium" Acta Cryst. Vol. 6, p. 753.
- 19. W. M. LOMER (1955) "The Beta-Alpha Transformation in Uranium-1.4 Atomic Per Cent Chromium Alloy" Inst. Metals London Monograph and Rept. Service Vol. 18, p. 243.
- 20. W. CHUBB (1953) "A Method of Measuring the Contribution of Crystal Structure to the Hardness of Metals" AEC Report BMI-X-112.
- 21. R. E. HUESCHEN and M. J. SANDERSON (1953) "Properties and Crystallography of Uranium Rolled in the Beta Phase" AEC Report HW-27082.
- 22. C. W. JACOP and B. E. WARREN (1937) "The Crystalline Structure of Uranium" J. Am. Chem. Soc. Vol. 59, p. 2588.
- 23. L. T. LLOYD and H. H. CHISWIK (1955) "Deformational Mechanisms of Alpha-Uranium Single Crystals" Jour. of Metals Vol. 7, p. 1206-1214.
- 24. R. W. CHAN (1953) "Plastic Deformation of Alpha-Uranium; Twinning and Slip" Acta Met. Vol. 1, p. 49-70.
- 25. E. A. CALNAN and C. J. B. CLEWS (1952) "The Prediction of Uranium Deformation Textures" Phil. Mag. Vol. 43, p. 93-104.
- 26. A. N. HOLDEN op. cit., p. 84-97.
- 27. H. AUBERT and J. GERLAND (1959) "Grain Refining of Uranium by Means of Small Additions and Heat Treatments" AEC Report HW-TR-24.
- 28. D. W. WHITE (1955) "Transformation Kinetics in Uranium-Chromium Alloys" Jour. of Metals Vol. 7, p. 1221.
- 29. J. BURKE and P. H. DIXON (1962) "The d ⇒ B Phase Transition in Pure Uranium" Jour. of Nuc. Materials Vol. 7, p. 38-45.

- 30. B. R. BUTCHER, et. al. (1961) "Grain Shapes in Quenched Uranium" Jour. of Nuc. Materials Vol. 4, p. 100-108.
- 31. R. B. RUSSELL and A. K. WOLFF(1963) "Summary Report on the Study of Beta Treatment of Uranium" AEC Report NML-2807.
- 32. R. KORNFELD (1959) "Structures of Beta-Quenched Uranium" AEC Report MCW-1429-Del., p. 145-155.
- 33. B. D. CULLITY op. cit., p. 124-128.

# APPENDIX

TABULATED VALUES OF  $\boldsymbol{\alpha}_{i}$  FOUND AT INDIVIDUAL SPECIMEN SURFACES

 $\boldsymbol{\varkappa}_{\mathtt{i}}$ 

O		(distance	e from	the q	luenched	surface	in	inches)
and comp.	(hk1)	<u>•011</u>	<u>•060</u>	<u>•097</u>	<u>•240</u>	<u>•494</u>		
1a	020	0.13	0.03	0.36	0.93	0.89		
.00 at.% Si	110	1.32	0•59	0.29	1 <b>.01</b>	0.63		
	021	0•38	0.43	0•74	2.06	3•23		
	002	1.32	2.12	2.35	2.63	1.36		
	111	1.76	0.87	0.47	0.80 .	0•75		
	112	1-41	2.04	1.31	0.87	0.96		
	130	0.12	0.04	0.19	0•79	1.59		
	131	0.33	0.12	0 <b>.</b> 27	0•53	<b>0.</b> 63		
	023	0.68	1•41	3.11	0.82	<b>0.7</b> 4		
	200	2.68	1.93	1.24	2.02	0•96		
	113	1.21	1.98	1.54	1.10	0.59		
	132	0.29	0.12	0.57	0.60	0.66		
	133	0.55	0•90	1.62	1.04	0.85		
	114	1.25	1.66	1.57	1.05	0.65		
	150	0.24	0.12	0.60	1.23	1.69		
	2 <b>23</b>	1.36	U <b>•</b> 90	0.65	1.01	0•94		
	152	0.30	0.14	0 <b>.</b> 14	0.61	1.25		
	312	2.42	2•36	0.92	0.57	0.51		

Specimen No.				- 1 4					
and comp.	(hk1)	•0 <b>0</b> 9	•030	<u>•046</u>	<u>•079</u>	<u>•101</u>	<u>•202</u>	<u>•312</u>	<u>•386</u>
1b 08 at % Si	020	0.19	0.07	0.00	0.09	0.21	0.47	1.58	1.20
•00 au• / D1	110	1.45	1.14	0.72	0.48	0.53	0•51	1.14	1.14
	021	0•Щ	0.34	0•27	0.64	0.36	0.81	0.67	0.44
	002	1.15	1.36	1.12	3•43	3.00	1.69	U•59	1.00
	111	1.62	1.45	1.61	1.17	0.93	0.52	0.62	0•71
	112	1.40	1.54	1.62	0•94	1.20	0•79	0.82	0.83
	130	0,29	0•28	0.00	0.07	0.08	0•47	1.02	1.19
	131	0.47	0.20	0.15	0.22	0•31	0.52	0.82	0.84
	023	0.71	0•75	<b>1.</b> 03	1.04	2.32	3.69	1.29	1.48
	200	2.38	2.86	1.68	1.00	0.97	1•49	0•94	0•78
	113	1.23	1.38	1.57	1.90	1.83	1.17	0.89	0.83
	132	0.44	0•45	0•32	0.55	0.51	1.57	0.87	0.95
	133	0.69	0.69	0.66	0.67	0.85	0.78	1.17	1.32
	114	1.10	1.26	1.84	2.23	1.98	1.47	1.06	0•77
	150	0.17	0.09	0.05	0.32	0•36	0.66	1.37	2.12
	223	1.52	1.43	1.78	0.89	1.17	0.77	0.50	0.67
	152	0.27	0.24	0.22	0.31	0.34	0.47	1.20	0•97
	312	2.19	2.45	2.34	1.97	1.09	0.43	1.33	1.08

Specimen No. and comp.	(hkl)	•011	•031	•047	•078	•095	<b>•1</b> 80	•273	•390
2a	020	0.20	0.11	0.09	0.00	0.13	0.30	0.49	0.74
•16 at•% Si	110	1.56	1.23	1.00	1.02	1.00	0.98	0.97	1.07
	021	0.47	0.36	0.35	0.52	0.69	1.01	1.52	1.37
	002	1.29	1.59	1.42	3•51	2•37	<b>2.5</b> 5	2.61	1.86
	111	1.74	2.06	1.82	2.04	1.58	1.12	1.06	0.95
	112	1.20	1.31	1.92	1.29	1.01	0.95	0.78	0.87
	130	0.24	0.14	0.24	0•19	0.40	0.70	1.04	1.71
	131	0.37	0.26	0.24	0•33	0.61	0.61	0.74	0.99
	023	0•92	0.95	0.91	0.68	1.09	1.09	1.69	1.21
	200	2.13	1.73	2•16	1.53	1.14	0.54	0.41	0.72
	113	1.10	1.27	1.42	1.45	1.52	1.54	0.82	0.77
	132	0.54	0.47	0.35	0.39	0.67	0•71	0.61	0.69
	133	0.71	0.63	0.57	<b>0.</b> 84	0.89	1.22	0.90	0.65
	114	0•90	1.24	1.34	1.59	1.72	1.48	0 <b>•9</b> 9	0.89
	150	0.20	0.11	0.09	0.22	0.54	0•79	0.84	1.24
	223	1.38	1.57	1.60	1.19	1.06	0.89	0.82	0.70
	152	0.40	0.21	0.22	0.31	0.56	0.55	0•99	1.10
	312	2.20	2.12	2.02	1.26	0•91	0.91	0.80	0.95

	60

and comp.	(hk1) 020	<u>•013</u>	<u>•028</u>	•0 <u>1</u> 8	<u>•073</u>	<u>109</u>	•168	<b>287</b>	<b>-</b> 480
<b>2</b> Ъ	020	• • •					بجهان فكعداوهم	<u>منہ مح</u> قد	
2b •16 at•% Si		0.10	<b>0.0</b> 6	0.02	0.04	0.16	0.16	0•39	0.92
•10 200/0 51	110	1.44	1.12	0.80	Ŭ <b>₀</b> 56	0.57	0.60	1.13	1.03
	021	0.30	0•26	<b>0</b> •18	0•29	0.50	0.48	1.10	1.05
	002	0•79	1.03	1.15	1.25	2.45	3•18	2•53	1.33
	111	1.63	1.44	1.63	1.63	0.81	0.76	0.87	0.90
	112	1.53	1.91	2.50	1.92	0.87	0.91	0.75	U•93
	130	0.11	0.00	0.04	0.03	0.23	0.56	0.94	1.56
	131	0.30	0.20	0•20	0.25	0.30	0.69	0.80	0.84
	023	<b>0.9</b> 8	1.06	0.80	1.36	3.40	2.08	0.67	0.95
	200	2.05	1.03	0•98	0.61	0.47	0.39	0.60	1.22
	113	1.30	2.10	2.08	1.99	1.65	1.08	0.77	0•79
	132	0•39	0.23	0•38	0•70	0.48	0.54	0.80	0•90
	133	0.65	0.70	0.61	0.70	0.76	0.92	1.29	1.23
	114	1.47	1.74	<b>1.9</b> 3	1.88	2.23	2.55	1.12	0•98
	150	0.21	0.11	0.09	0.09	0.16	0.36	1.82	1.18
	223	1.44	1.36	1.61	1.80	0.55	0•79	0.76	0.84
	152	0.24	0.22	0.20	0.49	0.62	<b>0.7</b> 8	1.01	0•93
	312	2.21	2.15	1.81	1.32	0•93	0.77	<b>0.9</b> 8	0.90

61

Specimen No. and comp.	(hkl)	<u>•010</u>	<u>•029</u>	<u>•045</u>	<u>•075</u>	<u>•098</u>	<u>.186</u>	<u>•286</u>	<u>•403</u>
3a	020	0.28	0.13	0.13	0.06	0.04	0.47	0•95	1.51
•51 at. % 51	110	1.74	1.71	1.60	1.72	2.04	0.78	0.74	0.80
	021	0.40	U•29	0.25	0.26	0.30	0•59	0.81	1.50
	002	0•79	U•99	0.95	1.30	1.65	2.07	1.63	1.99
	111	1.57	1.68	1.67	1.43	1.11	0.84	<b>∪</b> •70	0.86
	112	1.01	1.14	1.28	1.22	1.11	0.98	1.05	0.84
	130	0•46	0.21	0.22	0.23	0.08	0.55	1.14	1.03
	131	0.50	0.28	0.30	0.27	0.27	0.68	1.02	1.12
	023	0.85	0.87	1.06	0.96	1.03	1.17	1.32	1.21
	200	3.28	3.03	2.88	2.44	1.42	0.59	0•58	0•70
	113	0 <b>₊9</b> 2	1.06	1.13	1.36	1.38	1.65	1.34	1.12
	132	0.63	0.49	0.37	0.63	0.36	0•74	1.05	<b>0.9</b> 8
	133	0.66	0.54	0.57	0.58	0.66	1.78	1.19	<b>U</b> _88
	114	0•79	1.08	1.01	1.51	2.08	1.96	1.37	1.30
	150	0•36	0.34	0.15	0.21	<b>U</b> •16	0.68	0 <b>•9</b> 9	0.80
	2 <b>23</b>	1.14	1.20	1.14	1.09	1.34	0.85	0.85	0•77
	152	0.47	0.35	0.28	0.19	0.26	0.84	1.05	0.86
	312	2.25	2.43	2.61	2.21	1.86	0.42	0.45	0.40

•

		(	52

Specimen No.									
and comp.	<u>(hkl)</u>	•014	<u>•028</u>	•048	<u>•072</u>	<u>•102</u>	<u>•161</u>	<u>.280</u>	<u>.484</u>
3b	020	0.24	0.16	0.14	0 <b>.10</b>	0.12	0•17	1.40	0.82
	110	1.74	1.64	1.62	1.44	1.31	0.76	0.64	0•93
	021	0•43	0.43	0.37	0.32	0.37	<b>∪</b> •77	1.00	0•96
	002	1.53	1.40	1.49	2 <b>•1</b> 6	2.55	3.49	1.65	1.27
	111	1.66	1.74	1.74	1.74	1.53	1.05	0.82	0.94
	112	0,88	0.98	1.11	0.95	0.93	0.98	0.83	<b>u</b> •78
	130	0.27	0.34	0.27	0.18	0.11	0.31	1.05	1.26
	131	0.42	0.31	0.28	0.22	0.27	0.47	0.78	<b>∪</b> •86
	023	0•79	0.85	0.74	0.67	1.02	1.12	1.38	1.07
	200	3.01	3.21	3.46	2.44	2.07	0.95	0.40	0.55
	113	0.90	0.92	1.08	1.37	1.61	1.73	<b>1.</b> 03	1.04
	132	0.46	0.45	0.29	0.37	0.30	0.59	0.81	0.94
	133	0.56	0•55	0.54	0.65	0•79	1•41	1.32	1.09
	114	0.83	0•97	1.07	1.40	1.73	1.99	1.52	1.19
	150	0.44	0•39	0.28	0.20	0.15	0.40	0•97	1.36
	223	1.33	1.32	1.69	1.72	1.67	1.29	0•98	0.84
	152	0.47	0.36	0.30	0.23	0.22	0.31	1.11	1.12
	312	2.28	2.25	2.09	2.02	1.50	0.66	0.47	0.90

Specimen No.						
and comp.	(hkl)	<u>•010</u>	•059	<u>•097</u>	<u>•255</u>	<u>•493</u>
La Oli at d Co	020	0.16	0.05	0 <b>•</b> 08	0.96	0.48
	110	1.37	0.82	0.89	0.38	0.56
	021	0.26	0.12	0•16	0.84	3.69
	002	0.68	0.36	0.43	1.37	3.32
	111	1.56	1.77	1•73	0.54	0.67
	112	1.40	1.76	1.60	0•72	0.43
	130	0.46	0.16	0.12	0.28	0.41
	131	0.45	0•16	0•18	0.31	0.62
	023	0.97	1.03	2.21	5.40	0.95
	200	2.16	1.10	0.60	0.57	0.44
	113	1.48	2.92	2.69	1.28	0•78
	132	0.64	0.23	0•31	0.95	0.91
	133	0.70	0.50	0•58	1.05	1.21
	114	1.14	2.11	1.91	1.27	1.06
	<b>1</b> 50	0.24	0.01	0.12	0.28	0.59
	223	1.53	1.83	1.98	0.58	0.55
	152	U•29	0.14	0.09	0.38	1.19
	312	2.03	1•74	1.07	0.23	0.37

Specimen No.									
and comp.	(hk1)	<u>•012</u>	<u>•026</u>	<u>•045</u>	<u>•069</u>	<u>•101</u>	<u>•163</u>	<u>•284</u>	<u>•499</u>
4b	020	0.17	U <b>•</b> 07	0.03	U <b>•1</b> 0	<b>U</b> •13	1.18	3•98	0.62
	110	1.31	1.01	0.82	0.74	0•58	U•57	Ú <b>•7</b> 1	0 <b>.</b> 44
	021	U•33	0.24	0.21	0.20	0.40	0.72	0.93	1.99
	002	1.69	1.26	1.05	1.82	4.45	7•43	1.67	2.30
	111	1.27	1.25	0 <b>•9</b> 6	1.05	0•95	0.62	0.66	<b>u.</b> 66
	112	1.45	1.72	2.26	2.28	1.55	0.86	0.63	1.03
	130	0.24	<b>Q.1</b> 6	U•22	0.10	0.09	0.43	0.97	U•62
	131	0.30	0.24	0 <b>.</b> 17	0.20	0.23	0.38	0.89	0.66
	023	0.59	0.52	0.39	0.60	0.93	1.80	2.27	1.59
	200	4.00	3.91	4.38	3•52	2.55	1.64	1.24	0.60
	113	1.03	1.10	1.11	1.40	1.16	0•77	U_60	1 <b>.1</b> 8
	132	0.48	0.36	0.23	u <b>.</b> 18	0.35	0.54	0•78	1.07
	133	0.64	<b>u</b> •58	0.48	0.46	0•53	U•79	0.64	1.33
	114	0.76	0.71	0.73	0.95	0.87	0.93	0.49	<b>0.7</b> 8
	150	0.25	0•16	0.13	0.16	0.33	1.15	1.30	1.16
	2 <b>23</b>	1.20	1.41	1.35	1.22	0.89	0.81	0.80	0.60
	152	0.22	0.16	0.16	0.16	0.31	0.41	1.04	0.96
	312	2.90	3•47	3.87	3•37	2 <b>•9</b> 6	0.70	<b>U_5</b> 8	u <b>.</b> 60
		65							
--	--	----							

Specimen No.									
and comp.	(hkl)	<u>•011</u>	<u>•030</u>	<u>•046</u>	<u>•077</u>	<u>•103</u>	<u>•191</u>	<u>•291</u>	<u>•404</u>
5a	<b>U20</b>	0.28	0.21	0.23	0.20	0.44	2.19	1.17	1.25
	110	1.80	1.53	1.29	1.02	0.90	0.97	1.29	1.16
	021	0.35	0.30	0.32	0.50	1•38	2.53	0.74	1.16
	002	0•70	0.53	0.58	0.65	1.18	2.42	1.82	1.52
	111	1.48	1.37	1.21	<b>0</b> •86	0.65	<b>0.7</b> 4	0•99	1.26
	1 <b>1</b> 2	U•87	0.82	0.91	1.28	1.08	0.63	0.80	0.89
	130	0.25	0.36	0.13	0.38	0.13	0.82	0,90	1.05
	131	<b>0.36</b>	0.29	0.24	<b>0</b> •28	0.43	0.44	0•98	1.04
	023	0 <b>•9</b> 2	<b>0.9</b> 2	1.00	0•94	1.12	0.95	0.82	1.13
	200	5.23	8.49	8.40	5•78	1.91	1.02	1.18	1.03
	113	0.81	0.89	1.11	1.57	1.49	0.68	0.80	0.89
	132	0 <b>.</b> 46	0•30	0.24	0.48	0.36	0.61	0•94	0.77
	133	0•52	0•39	0.43	0•59	0.84	0.83	1.01	0.78
	114	0.80	0•77	1.07	1.30	1.06	0.83	1.27	0.82
	150	0.39	0.35	0•39	0.15	0.47	1.72	0.87	1.02
	223	0.96	0.87	0.84	1.18	1.25	0.76	0.75	<b>0•</b> 82
	152	0.35	0•2li	0.21	0.32	0.65	1.02	0.89	0.96
	312	2.53	2.44	2.40	2.24	2.28	0,50	1.09	0.86

		6	6

Specimen No. and comp.	<u>(hkl)</u>	<u>•015</u>	<u>•028</u>	<u>•049</u>	<u>•071</u>	<u>•103</u>	<u>•166</u>	<u>•283</u>	<u>•489</u>
5b	020	0•10	0.06	0.06	0.04	0.00	0.53	3.76	1.35
•11 at•% ur	110	1.71	1.54	1.30	0.85	0•53	0•49	0•98	0.54
	021	0.28	0•20	0•16	0.17	0.24	1.01	0.61	0.93
	002	0.80	0.55	0.60	0,63	1.50	2.41	0.83	0.47
	111	1.48	1•46	1.48	1.49	1.49	0.48	0.56	0•91
	112	1.36	1.31	1.17	0.83	0.60	0.55	0.43	0.78
	130	0 <b>.1</b> 8	0.29	0.11	0.10	0.07	0.20	0.51	0•73
	131	0.33	0.27	0.17	0.11	0.09	0.18	0•55	0•73
	023	0.60	0.50	0.61	0.66	0.74	0.95	2.02	2.45
	200	2.10	2.08	1.40	0.76	0.60	0 <b>.9</b> 3	2•30	2.83
	113	1.34	1.49	2.49	2.88	2.56	<b>2.0</b> 2	0•99	1.11
	132	0.37	0.37	0.24	0.28	0.25	0.29	0•91	1.00
	133	0.71	0.70	0.52	0.54	0.64	1.11	0.97	0.90
	114	1.28	1.68	2.77	4.09	5.23	4.36	1.37	1.16
	150	0•18	0 <b>.</b> 16	0.07	0.03	0.06	0.58	0 <b>.</b> 68	0•94
	223	1.30	1.16	0•77	0.60	0.35	0.35	0.47	0.89
	152	0.41	0.32	0.22	0.13	0,20	0•74	0•92	0.96
	312	2•53	2.70	2 <b>.30</b>	1.75	<b>0.9</b> 8	0.39	0.74	0.37

Specimen No.				- • • •					1 0
and comp.	(hk1)	<u>•013</u>	<u>•026</u>	<u>•045</u>	<u>•071</u>	<u>•102</u>	<u>•158</u>	<u>•278</u>	<u>•478</u>
6a 33 at % Cr	020	0.26	0.11	0.05	<b>0.</b> 05	0.06	0.26	0.12	1.11
	110	2 <b>.09</b>	2.15	1.93	1.52	1.39	0•74	0.62	1.78
	021	0•39	0.25	0•19	0.17	0.25	0•37	0.37	0.44
	002	1.11	1.01	0.90	0.67	1.34	3•39	3.42	1.13
	111	1.35	1.23	1.18	1.33	1.56	1.07	0.90	0.62
	112	0.83	0.65	0.57	0.40	0.32	0.42	0.60	0.53
	130	0.14	0.07	0.20	0.07	0 <b>.1</b> 8	0.16	0.27	0.52
	131	0.32	0•26	0.24	0.27	0.26	0•20	0.46	1.07
	023	0•96	0.94	0.88	0.82	1.17	3.27	1.63	0.76
	200	3.57	3•36	2.62	2.57	2.81	1.50	0 <b>.</b> 50	0.23
	113	0.78	0.80	0.81	0•74	0.62	1.07	1.50	1.08
	132	0.49	0.29	0.35	0.38	<b>0.3</b> 8	0.67	1.04	2.00
	133	0.44	0.41	0.37	0.30	0.34	0.44	1.32	1.46
	114	0•91	1.04	1.09	1.37	1.33	2.04	2 <b>.</b> 57	1.05
	150	0.26	0.19	0.12	0•14	0.17	0.16	0.30	0.89
	223	0.82	0•70	0.56	0.45	<b>0.</b> 38	0.31	0.40	0.48
	152	0.34	0.28	0.23	0.24	0.24	0.29	0•72	1.57
	312	2 <b>.91</b>	3•59	4.28	4.64	3•99	1.59	0.80	0.62

Specimen No. and comp.	<u>(hkl)</u>	<u>•014</u>	<u>•027</u>	<u>•047</u>	<u>•070</u>	<u>•099</u>	<u>•155</u>	<u>•274</u>	<u>•495</u>
6b •33 at.% Cr	020	0.20	0.02	0.04	0.04	0.00	0.02	0.26	1.03
	110	1.47	1.12	0.86	0.43	0•39	0.30	0.41	1.07
	021	0.31	0.20	0.19	0.15	0.12	0.15	0.28	<b>0•</b> 58
	002	1.07	0.89	0.56	0.60	0•59	1.07	9.14	1.59
	111	1.84	1.82	2.19	2.58	2 <b>•51</b>	1.70	0.29	1.14
	112	1.39	1.91	2.19	2.82	2.27	1.29	0.37	0.52
	130	0.41	0.32	0.19	0.19	0.08	0.13	0.00	0.43
	131	0.47	0•39	0.35	0.24	0.27	0.28	0.11	0.47
	023	0•77	0.88	0•92	0.87	1.19	3.87	3•33	1.89
	200	2.20	2.49	2.55	1.74	1.02	0•92	0.36	2.86
	113	1.23	1.70	1.98	2.65	2.70	1.78	1.35	0.63
	132	0.45	0•44	0.40	0.18	0.37	0.30	0.29	1.40
	133	0.58	0.43	0•37	0.40	0.54	0.56	0•33	1.54
	114	1.13	1.20	1.42	1.44	1.80	1.93	3.03	0.71
	150	0.26	0.16	0 <b>.09</b>	0,00	0.05	0.12	0.14	0.51
	223	1.40	1.58	1.69	1.94	2 <b>.1</b> 0	1.61	0.24	0.57
	152	0.33	0•18	0.10	0•13	0.10	0.16	0.16	0•88
	312	2.11	2.07	1.71	1.25	1.00	0.74	0.40	0.92

## VITA

The author was born on July 31, 1940, near Malden, Missouri. He received his primary and secondary education in Fornfelt (now Scott City), Missouri. He entered college at the Southeast Missouri State College, Cape Girardeau, Missouri, in September, 1958. He enrolled at the Missouri School of Mines and Metallurgy in September, 1960 and received a Bachelor of Science Degree in Metallurgical Engineering-Nuclear Option from this institution in January, 1963.

He was enrolled as a regular graduate student at the Missouri School of Mines and Metallurgy during January through May of 1963. He was employed by the Mallinckrodt Chemical Works, Uranium Division, Weldon Springs, Missouri, in June, 1963. He worked for this company for 15 months , during which time he was involved in basic physical metallurgical research. He returned to school at the Missouri University at Rolla in September, 1964. From September, 1964 to January, 1965 the author held the Kroll Fellowship from the Metallurgical Engineering Department.