212
-16-86
JAH

Y-12

OAK
RIDGE
Y-12
PLANT

UNION
CARBIDE

OPERATED BY
UNION CARBIDE CORPORATION
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

MASTER

R-1244

Y-2213

LOW ALLOY ADDITIONS OF IRON, SILICON, AND
ALUMINUM TO URANIUM – A LITERATURE SURVEY

R. L. Ludwig

December 1980

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price codes
Printed copy: A03
Microfiche copy: A01

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Date of Issue: **December 31, 1980**
Distribution Category: UC-25

Y-2213

LOW ALLOY ADDITIONS OF IRON, SILICON, AND ALUMINUM TO URANIUM – A LITERATURE SURVEY

R. L. Ludwig
Metals and Ceramics Department
Y-12 Development Division

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Oak Ridge Y-12 Plant
P. O. Box Y, Oak Ridge, Tennessee 37830

Prepared for the Department of Energy
Under US Government Contract W-7405-eng-26

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

See

ABSTRACT

A survey of the literature has been made on the experimental results of small additions of iron, silicon, and aluminum to uranium. Information is also included on the constitution, mechanical properties, heat treatment, and deformation of various binary and ternary alloys.

CONTENTS

SUMMARY	4
INTRODUCTION	5
A LITERATURE SURVEY OF ALLOY ADDITIONS TO URANIUM	7
Constitution	7
Uranium-Iron Binary Alloy Phase Diagram	7
Uranium-Silicon Binary Alloy Phase Diagram	10
Uranium-Iron-Silicon Ternary Alloy Phase Diagram	11
Uranium-Aluminum Binary Alloy Phase Diagram	13
Uranium-Aluminum-Iron Ternary Alloy Phase Diagram	14
Uranium-Silicon-Aluminum Ternary Alloy Phase Diagram	19
Mechanical Properties	19
Heat Treatment	24
Microstructural Changes Resulting from Heat Treatment	24
Effect on Alloys of Varying Heat-Treating Parameters	25
Effect of Heat Treatment on Properties	26
Effects of Alloy Additions on Recrystallization	27
Time-Temperature-Transformation Characteristics	27
Deformation	31
REFERENCES	32

SUMMARY

Research efforts in uranium metallurgy in the late 1950s and 1960s were directed toward the dimensional stabilization of uranium reactor fuel elements operating at elevated temperatures. Although high-temperature properties of the elements were of primary interest, information was developed and published which is currently applicable to the improvement of their room-temperature properties through small additions of iron, silicon, and aluminum.

Of significance among the data derived from the literature are the constitution diagrams for the high uranium end of the U-Fe, U-Si, and U-Al binary systems. Also presented are the phase relationships existing in isothermal sections of the uranium corner of the U-Fe-Si and U-Fe-Al ternary systems. Occasional references to room- and elevated-temperature mechanical properties were also encountered, and these data are included to show the general effects of small additions of iron, silicon, and aluminum. In most cases, however, there was no attempt to control influential variables such as hydrogen in the uranium; so there is some question that the effects are solely due to the alloy additions.

Considerable information was available in the literature pertaining to the development of heat treatments which would most nearly produce the desired stabilities of the operating uranium fuel elements. These principally involved quenching from the beta or gamma regions followed by a high-alpha precipitation treatment. Microstructures produced by these various treatments were the subject of studies which attempted to show the roles of the various alloying additions with respect to solution time, precipitate size, and alpha grain size. The effect of the quenching rate on the microstructure was also the subject of several investigations—particularly with regard to its effect on the refinement of the alpha grain size, the production of bainitic and martensitic structures, and crystallographic texture. The relationships between various heat treatments and hardness had been investigated as was the correlation between recrystallization and alloy content. Lastly, time-temperature-transformation relationships have been reported for several alloys of interest.

The influence of the alloy content on deformation is a subject of current interest and about which very little information was found. Uranium was shown to be capable of large amounts of strain hardening, and some data were found relating impurity content to separating force during the rolling operations.

INTRODUCTION

Fuel elements of pure uranium metal made for use in reactors in the 1950s were fabricated from castings which had been wrought into rods by extrusion or rolling. Slugs machined from these rods were metallurgically bonded within aluminum cans to promote heat transfer and to prevent corrosion of the uranium and contamination of the reactor cooling water. Early operating experience disclosed that the fuel elements, under the influence of irradiation and the elevated-temperature thermal-cycling conditions which is prevalent in reactors, were undergoing severe dimensional distortion. The consequence of this distortion was blockage of the coolant passages in the reactor with resultant shutdown.

One form of dimensional distortion was warpage caused by relief of residual stresses; a second form was growth resulting from nonrandom crystallographic orientation within the fuel element. This orientation was a result of the working, which the fuel-element metal had undergone in reducing it from a casting to a rod. Highly crystallographically oriented elements which had been subjected to many thermal cycles have been shown to increase their original length as many as six times. A third form of dimensional distortion was manifested by bumping or pimpling the surface of uranium fuel rods which had been fabricated from cast uranium. This was a result of the effect of temperature cycling on the very coarse grain of the cast material.

For improved uranium fuel performance, it was soon determined that the crystal structure should be as randomly oriented as possible and should consist of fine grains. Beta quenching of uranium containing small concentrations of various-alloying additions was found to produce both a randomized crystal structure and a fine grain size. The British "adjusted uranium," the French Sicral alloys, and the United States alloys were all developed for this purpose; and all were based on the effects of a beta heat treatment on alloys containing increased amounts of impurities normally present in uranium, i.e., iron, silicon, aluminum, and (to a lesser extent) chromium. These elements were added in concentrations commonly ranging from 100 to 1000 ppm, and usually two or more were needed to obtain the desired properties.

Because the uranium fuel rods operated in the high-alpha-temperature range of uranium, properties of the various alloys at elevated temperatures were more interesting than were room-temperature properties; and, consequently, little ambient-temperature data were developed. However, considerable effort was expended to investigate the behavior of these alloys. Binary and ternary constitution diagrams were determined in order to establish the solubility limits of the alloying elements in the three uranium allotropes and to assist in establishing heat-treatment procedures.

The particular interest which prompted this literature investigation at the Oak Ridge Y-12 Plant^(a) was a desire to discover how the room-temperature mechanical properties of

(a) Operated by the Union Carbide Corporation, Nuclear Division, for the Department of Energy.

uranium could be improved by small additions of the impurity elements normally present in the metal. Much of the information which was applicable to the development of improved uranium fuel elements is also of potential value in the present endeavor, and an attempt has been made to include only that which is of such value. The discussion, therefore, has been divided into four sections: constitution, heat treatment, mechanical properties, and deformation. Each has direct application to the subject of interest.

A LITERATURE SURVEY OF ALLOY ADDITIONS TO URANIUM

CONSTITUTION

Work on the constitution of uranium alloyed with minor additions of aluminum, silicon, and iron was performed in the early and mid-1960s at Mallinckrodt,^(b) National Lead,^(c) and Nuclear Metals.^(d) The binary systems U-Al, U-Si, and U-Fe were studied in some depth, and isothermal sections of the uranium rich corner of the ternary systems were also investigated. The quaternary system was examined only superficially using optical microscopy.

An alloy-development summary report¹ broadly covers the extent of the investigations conducted in this report. In general, the solid-solubility limits were obtained for aluminum, silicon, and iron in the three uranium allotropes. Each system showed an increasing solubility with temperature, in each phase, for the additive element with a marked stepwise advance in solubility when going from the alpha to beta to gamma phases. The U-Si system was an exception in one respect; i.e., from beta to gamma, the solubility of silicon dropped to about one-half the level just above the transformation temperature that it had been just below. This is because of the peritectoid reaction in which:

Relatively large amounts of silicon and iron are soluble in the β -phase, but aluminum has a low solubility of about 100 ppm just above the $\alpha \rightarrow \beta$ transformation which increases to about 600 ppm just below the $\beta \rightarrow \gamma$. At the uranium end of the three binary systems, only the intermetallic compounds U_3Si , U_6Fe , and UAl_2 have been identified. The ternary systems showed no additional compounds; but there was some alloy solubility in the compounds, e.g., U_6Fe will dissolve about 0.5% silicon but essentially no aluminum. Similarly, U_3Si will dissolve about 0.3% aluminum but no iron; and UAl_2 will dissolve only a very small amount of silicon but about 2.25% of iron.

The determination of ternary diagrams was difficult and was not completed to the extent desired, but the binary diagrams have been very useful in evaluating alloying effects.

Uranium-Iron Binary Alloy Phase Diagram

The U-Fe phase diagram has been studied both at Mallinckrodt and Nuclear Metals. At Mallinckrodt, the boundaries and phase fields were determined in uranium alloys containing 400- to 5000-ppm iron by weight at temperatures from 600 to 1200°C.² The various alloy compositions are shown in Table 1; they were prepared by magnesium bomb reduction,

(b) Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Mo.

(c) National Lead Company of Ohio, Cincinnati, Ohio.

(d) Nuclear Metals Division of Textron, Inc., West Concord, Mass.

equilibrated 75 hours at temperature in vacuum, and water quenched.² Thermal arrests on heating resulting from phase transformations² are given in Table 2. The low heating rate was maintained to assure a minimal superheating effect. Figure 1 is the equilibrium phase diagram obtained.² The boundaries between the one- and two-phase fields were determined by electron-beam microprobe analysis of the matrix of the 3000 ppm sample equilibrated for 75 hours. The results appear in Table 3.

Table 1
COMPOSITIONS OF PURE URANIUM AND URANIUM-IRON ALLOYS

Specimen Number	Chemical Analysis (ppm) ⁽¹⁾	Spectrochemical Analysis (ppm) ⁽¹⁾							
	Carbon	Boron	Chromium	Copper	Iron	Magnesium	Manganese	Nitrogen	Silicon
Pure Uranium	24	< 0.10	< 4	4	18	8	2	5	16
Uranium-Iron 4		< 0.10	12	6	600	10	12	26	42
Uranium-Iron 6		< 0.10	12	6	800	< 10	12	25	48
Uranium-Iron 8		< 0.11	10	5	1000	10	7	22	44
Uranium-Iron 9		0.10	13	9	1200	16	11	30	51
Uranium-Iron 10		0.13	12	7	1550	11	12	32	62
Uranium-Iron 11		< 0.30	15	4	2100	30	15	19	100
Uranium-Iron 12		0.19	11	17	2400	24	16	28	71
Uranium-Iron 13		0.14	8	13	2600	21	8	29	57
Uranium-Iron 14		0.19	8	14	3000	14	9	30	65
Uranium-Iron 15		0.22	13	17	5000	12	10	44	100

(1) All values given in parts per million by weight.

It was also determined that alloys quenched from the gamma and beta fields will retain some beta phase which slowly transforms martensitically to alpha at room temperature. The retention of beta requires a certain minimum cooling rate, believed to be about 100°C/s.

Russell³ at Nuclear Metals performed less extensive work on the U-Fe binary system. The solid solubility of iron in uranium was measured using optical and electron microscopy, electron microanalysis, and electrical-resistance ratio measurements. The data obtained have been added to Figure 1 and are tabulated in Table 4. The data of Schierdin and Ferguson⁴ have been similarly treated in Figure 1 and Table 5. They also report that the maximum solubility of iron in alpha uranium is 180 ± 10 ppm at the eutectoid temperature of 667°C. The minimum solubility in the beta phase at 667°C is 340-ppm iron with the precision of this number being about ± 100 ppm. The maximum solubility of iron in beta uranium is about 990 ± 100 ppm, and the minimum solubility in the gamma phase at the eutectoid temperature of 763°C is about 1850 ± 150 ppm.

Swindells⁵ has published data on the solubility of iron in solid uranium. He reported 0.003-wt % iron soluble at 666°C, 0.085-wt % at 763°C, and 0.223- and 0.305-wt % at 780

Table 2
THERMAL ARRESTS OF PURE URANIUM AND URANIUM-IRON ALLOYS

Material	Heating (°C)		
	$\alpha \rightarrow \beta$	$\beta \rightarrow \gamma$	$\gamma \rightarrow \gamma + L$
Pure Uranium	672	770	-
1200 ppm Iron	667	763	-
3000 ppm Iron	668	764	850
5000 ppm Iron	667	761	805

Figure 1. THE URANIUM-IRON EQUILIBRIUM PHASE DIAGRAM.

Table 3
MICROPROBE ANALYSIS OF THE IRON CONTENT IN THE
GAMMA AND BETA URANIUM MATRICES

Material	Annealing Temperature (°C)	Annealing Time (h)	Iron Content in Uranium Matrix (ppm)
Uranium-Iron 14	797	75	2950 ± 5 to 10%
Uranium-Iron 14	780	75	2500 ± 5 to 10%
Uranium-Iron 14	774	75	2270 ± 5 to 10%
Uranium-Iron 14	750	75	845 ± 5 to 10%
Uranium-Iron 14	693	75	560 ± 5 to 10%
Uranium-Iron 14	690	75	550 ± 5 to 10%

and 800°C, respectively. The β -phase eutectoid point was 0.027 wt % at 661°C and that of the γ -phase was 0.15 wt % at 763°C. These data have also been shown in Figure 1.

Other authors have also contributed to an understanding of the U-Fe binary system. Paris⁶ determined the alpha-beta transformation temperatures for uranium alloys with 234- and 1032-ppm iron by differential thermal analysis. On heating, the transformation occurred at 676 and 677°C and on cooling at 621 and 625°C for the respective alloys. On a high-purity uranium standard sample, the equivalent temperatures were 674 and 645°C. Russian workers⁷ reported the solubility of iron in alpha-uranium at 600°C to be <0.01 at. % (<0.0024 wt %).

Uranium-Silicon Binary Alloy Phase Diagram

The constitution of high uranium-silicon alloys over the range of 30- to 3000-ppm silicon was investigated by Straatman and Neumann.⁸ The alloys were prepared by bomb reduction with silicon added as the element. The thermal arrests at phase transformations were determined by differential thermal analysis and are shown in Table 6. Substantial undercooling occurred

Table 4
SOLUBILITY DATA OF IRON IN
URANIUM FROM RUSSELL

Temperature (°C)	Iron in Uranium (ppm)
1100	1220
1000	2250
900	< 3085
825	≈ 3100
790	≈ 1800
750	< 790
725	680
700	425
635	59/12

Table 5
SOLUBILITY DATA OF IRON IN
URANIUM FROM SCHIERDING
AND FERGUSON

Temperature (°C)	Iron in Uranium (ppm)
550	113 ± 9
650	170 ± 13
700	460 ± 100
750	810 ± 85
790	2900 ± 150

Table 6
THERMAL ARRESTS AT PHASE TRANSFORMATIONS IN THE
HIGH URANIUM-SILICON ALLOY SYSTEM

	Heating (°C)		Cooling (°C)	
	$\alpha \rightarrow \beta$	$\beta \rightarrow \gamma$	$\gamma \rightarrow \beta$	$\beta \rightarrow \alpha$
Pure Uranium	673	775	774	661
Uranium-Silicon Alloys (650- to 3000-ppm Silicon)	665	785	777	624

in the $\beta \rightarrow \alpha$ transformation. Figure 2 is the phase diagram obtained in this investigation. It reveals that silicon depresses the α - β phase transformation slightly to 665°C and raises the β - γ transformation to 785°C. The maximum solubility of silicon in uranium in the α phase is 170 ppm. This increases to 1590 ppm in the β phase at 785°C. Because of the peritectoid decomposition of the γ phase, the solubility in the γ phase at 785°C decreases to 870 ppm. The maximum solubility of silicon in the γ phase is 2150 ppm at 925 and 985°C.

Figure 2. THE URANIUM-SILICON EQUILIBRIUM PHASE DIAGRAM.

Work by others has provided additional information on the uranium-silicon binary system. A Russian report⁷ indicates that the solubility of silicon in the α phase at 600°C is <0.085 at. % (<0.01 wt %). Paris,⁶ working with two uranium-silicon alloys containing 225- and 963-ppm silicon, respectively, confirmed the lowering of the $\alpha \rightarrow \beta$ transformation temperature. He reported a temperature of 669°C whereas Table 6 shows 665°C. For the $\beta \rightarrow \alpha$ transformation, the temperatures for the two alloys were 621 and 600°C, lower than the 624°C reported in Table 6. It was concluded that a lower cooling rate would have raised the 600°C temperature to approximately 621°C.

Uranium-Iron-Silicon Ternary Alloy Phase Diagram

Workers at Mallinckrodt⁴ determined isothermal sections of the uranium-iron-silicon ternary phase diagram in connection with work on alloys with enhanced radiation stability.

Electron-beam microprobe studies were made on as-quenched alloy samples, assuming that they represented the structure at the temperature from which they were quenched. The alloys were prepared by bomb reduction with the alloying additions being made as elements. The allotropic-transformation temperatures were determined by differential thermal analysis and appear in Table 7. There is relatively little effect of the alloy additions on these temperatures.

The solubility limits of iron and silicon in the uranium matrix and in the U_6Fe and U_3Si intermetallic compounds^{4,9} are shown in Tables 8 and 9. The effect of the β - γ peritectoid reaction in the uranium-silicon system is apparent in the ternary data in that the silicon solubility does not change significantly in going from 750 to 790°C compared to the large change in iron solubility. Silicon is soluble in U_6Fe to about

Table 7
ALLOTROPIC TRANSFORMATION TEMPERATURES
FOR THE URANIUM-IRON-SILICON TERNARY
ALLOY SYSTEM⁽¹⁾

Material	$\alpha \rightleftharpoons \beta$ (°C)	$\beta \rightleftharpoons \gamma$ (°C)
Pure Uranium	668	771
0.3 wt % Silicon	665	785
1.63 wt % Silicon		
0.81 wt % Iron	666	782
0.53 wt % Silicon		
0.27 wt % Iron	663	780
0.67 wt % Silicon		
1.10 wt % Iron	665	780
0.26 wt % Silicon		
2.0 wt % Iron	665	771
0.26 wt % Iron	667	763

(1) Precision is $\pm 2^\circ C$.

Table 8
THE SOLUBILITY LIMITS OF IRON AND SILICON IN THE URANIUM
MATRIX OF THE URANIUM-IRON-SILICON TERNARY ALLOY
SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE

Annealing Temperature (°C)	Iron Solubility (ppm)	Silicon Solubility (ppm)
650	120 $\pm$ 50	-
700	350 $\pm$ 30	990 $\pm$ 100
750	570 $\pm$ 50	1440 $\pm$ 100
790	1540 $\pm$ 180	1460 $\pm$ 170

Table 9
THE SOLUBILITY LIMITS OF IRON AND SILICON IN U_6Fe AND U_3Si IN
URANIUM-IRON-SILICON TERNARY ALLOY SYSTEM AS
DETERMINED WITH THE ELECTRON MICROPROBE

Annealing Temperatures (°C)	U_6Fe		U_3Si
	Iron (wt %)(1)	Silicon (wt %)	Iron (wt %)
538	2.76(2)	0.51(2)	-
649	2.77(2)	0.55(2)	-
700	2.90	-	0.2
732	2.93(2)	0.43(2)	-
750	2.84	-	0.4

(1) The precision of the average at the 95% confidence level is ± 0.03 wt %.

(2) These data were reported previously by Ferguson et al.

0.5 wt % at the alpha temperatures, decreasing to about 0.4 wt % at the beta temperature. The iron concentration in U_6Fe correspondingly increases by about the same amount. Iron is only slightly soluble in U_3Si . Ternary isothermal sections derived from these data and Schierding and Ferguson's⁴ data in Figure 1 are shown in Figures 3 and 4.

Figure 3. THE 700 AND 750°C ISOTHERMAL SECTIONS OF THE URANIUM-IRON-SILICON TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Conclusions from Reference 4 are that the solubility limits of iron and silicon do not differ appreciably from those of their binaries. The same intermetallic phases present in the binary systems, U_6Fe and U_3Si , were the only ones found in these dilute ternary alloys. The $\gamma \rightarrow \beta$ transformation temperature is not significantly changed by the addition of the third element to either the U-Fe or U-Si binary system; however, the $\beta \rightarrow \gamma$ temperature is dependent on the ternary composition.

Uranium-Aluminum Binary Alloy Phase Diagram

The solubility of aluminum in uranium has been investigated by Russell¹⁰ and by Schierding and Ferguson¹¹ in connection with their work on the ternary uranium-iron-aluminum diagram. Figure 5 is from Russell¹⁰ and represents not only his work but that of others who had previously done work in this area. Solubility limits from the report of

Figure 4. THE 790°C ISOTHERMAL SECTION OF THE URANIUM-IRON-SILICON TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Schierding and Fergason¹¹ also have been added to the figure. Tables 10 and 11 contain the data from References 10, 11, and 12. In other work, Reference 7, the solubility of aluminum in uranium was determined to be 0.0011 wt % at 600°C. Also, for aluminum contents of 211 and 974 ppm, the $\alpha \rightarrow \beta$ transformation temperatures were 675 and 678°C, respectively; and the $\beta \rightarrow \gamma$ temperatures were 634 and 636°C.⁶

Uranium-Aluminum-Iron Ternary Alloy Phase Diagram

Since the ternary diagram work by Schierding and Fergason¹¹ followed that of Russell¹⁰ and includes it, only the later work will be reviewed. Their alloys were prepared by bomb reduction with the alloying materials added as the elements. One of the difficulties encountered was that the UAl_2 was distributed so finely that it is uncertain whether the uranium matrix areas (analyzed by the electron microprobe for aluminum solubility determinations) were free of UAl_2 . Table 12 shows the measured solubility limits of iron and aluminum in the ternary alloy at various temperatures.¹¹ Figures 6 through 10 are isothermal sections at various temperatures.^{10,11} Russell's¹⁰ data at 635°C are shown in Figure 6 on the Schierding and Fergason¹¹ 650°C isotherm. The solubility limits are much higher than Russell's, suggesting that submicroscopic iron and aluminum precipitates may have been present in their microprobe samples.

Figure 5. URANIUM-ALUMINUM CONSTITUTION DIAGRAM.

Table 10
SOLUBILITY LIMITS OF ALUMINUM IN URANIUM
DETERMINED BY OPTICAL AND ELECTRON
MICROSCOPY ELECTRON MICROANALYSIS,
AND ELECTRICAL-RESISTANCE-RATIO
MEASUREMENTS

Temperature (°C)	Solubility (ppm)
635/550	44/35
750/700	263/162
790	1550
850	> 2500
925	> 2500
1000	5000/2000

Table 11
SOLUBILITY LIMITS OF ALUMINUM IN THE URANIUM
MATRIX OF THE URANIUM-ALUMINUM BINARY
ALLOY SYSTEM AS DETERMINED WITH THE
ELECTRON MICROPROBE

Annealing Temperature (°C)	Aluminum Solubility (ppm)
650	≈ 160
700	460 ± 130
724	≈ 440
752	≈ 530
765	1710 ± 10-20%
805	2180 ± 10-20%
822	2330 ± 10-20%
830	2115 ± 70
890	3050 ± 10-20%

The solubilities of iron and aluminum in U_6Fe and UAl_2 in the U-Fe-Al ternary system¹³ are given in Table 13. Aluminum solubility in U_6Fe is very small (< 0.03 wt %). The iron solubility in UAl_2 is very large, increasing with temperature, while the aluminum concentration in UAl_2 correspondingly decreases with increasing temperature.

Table 12

SOLUBILITY LIMITS OF IRON AND ALUMINUM IN THE URANIUM MATRIX OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE

Annealing Temperature (°C)	Iron Solubilities (ppm)	Aluminum Solubilities (ppm)
650	-	≈ 140
700	≈ 340	335 ± 120
750	910 ± 30	≈ 720
790	2900 ± 850	2380 ± 100
830	2870 ± 70	3030 ± 160

Table 13

THE SOLUBILITY LIMITS OF IRON AND ALUMINUM IN U_6Fe AND UAl_2 IN THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE

Annealing Temperature (°C)	U_6Fe		UAl_2	
	Iron (wt %)(1)	Aluminum (wt %)	Iron (wt %)(1)	Aluminum (wt %)
550	3.75		2.02	
650	3.77(2)	< 0.03	2.29	16.8(2)
700	3.69		2.71	
750	3.71		3.34	16.1(2)

(1) The precision of the average at the 95% confidence level is ± 0.03 wt %.

(2) These data were reported by Ferguson and Neumann.

Figure 6. THE 650°C ISOTHERMAL SECTION OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Figure 7. THE 700°C ISOTHERMAL SECTION OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Figure 8. THE 750°C ISOTHERMAL SECTION OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Figure 9. THE 790°C ISOTHERMAL SECTION OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Figure 10. THE 830°C ISOTHERMAL SECTION OF THE URANIUM-IRON-ALUMINUM TERNARY ALLOY SYSTEM AS DETERMINED WITH THE ELECTRON MICROPROBE.

Uranium-Silicon-Aluminum Ternary Alloy Phase Diagram

No data were found describing the high uranium corner of the uranium-silicon-aluminum ternary diagram.

MECHANICAL PROPERTIES

Since the emphasis for the development of high uranium alloys arose from the need for nuclear fuel that would resist distortion at high-temperature and radiation exposures, there was little reason to measure room-temperature mechanical properties. Elevated-temperature creep strength was believed to be a property which was a measure of an alloy's usefulness for reactor application. Because of the long duration of creep tests, hot-hardness measurements were tried as a short-time substitute test; however, hot hardness did not prove to be a reliable indicator of long-term high-temperature flow resistance.

Some impact and tensile tests were made to compare alloys and unalloyed uranium. Over the range -73 to 177°C , little change in impact properties was observed; all specimens were brittle at temperature below 93°C and tough and ductile at higher temperatures. Samples of beta oil-quenched alloy seemed superior to beta water-quenched alloy, suggesting microcracking as a result of the more drastic quench. Tensile tests showed the same kind of variability, all materials being relatively brittle below 93°C and ductile above. Iron appeared to definitely lower ductility and, in amounts greater than 200 ppm, decreased ductility at all alpha temperatures.¹

Property data on other alloys were apparently clouded by the possibility that hydrogen may have had a detrimental effect which obscured the true effect of alloying.¹ This view is furthered by a literature survey published in 1969¹⁴ in which the effects of grain structure, grain size, strain rate, processing, test conditions, and metallic impurities on the low-temperature ductility of uranium were reviewed. It was suggested that previous studies of impurity effects at low temperatures are of questionable value because the thermal treatments did not exclude hydrogen as a variable. Also, it was pointed out that no systematic study of the effect of specific metallic impurities on the low-temperature properties of vacuum-annealed uranium has been made.

An English report¹⁵ published in 1970 attempted to make a systematic study of impurities on the properties of wrought uranium, but the annealing environment was not mentioned. The question as to whether hydrogen was removed from the test specimens is, therefore, unresolved. Figures 11 and 12 are from this report and illustrate the effect of additions of iron, silicon, and aluminum on the room-temperature properties of reactor-grade uranium. It was also reported that iron in excess of 550 ppm and silicon and aluminum additions between 1000 and 2000 ppm caused weld cracking.

Elevated temperature tensile tests on cast uranium base alloys containing small additions of aluminum, carbon, iron, or silicon were conducted by National Lead.¹⁶ Figures 13 and 14 summarize the mechanical property data obtained from these tests. The composition of the

Figure 11. THE EFFECT OF IRON-, SILICON-, AND ALUMINUM-ALLOY ADDITIONS ON THE TENSILE STRENGTH OF URANIUM.

Figure 12. THE EFFECT OF IRON-, SILICON-, AND ALUMINUM-ALLOY ADDITIONS ON THE DUCTILITY OF URANIUM.

unalloyed control sample referred to in the figures was 68-ppm carbon, 62-ppm iron, 28-ppm silicon, and 18-ppm aluminum.

Room-temperature tensile data were also obtained at the Oak Ridge Y-12 Plant¹⁷ as part of an effort to characterize the effect of various contaminants on the uranium metal stream. Wrought alloys containing low levels of nickel-chromium (in a ratio of 8 to 18 by weight), silicon, and iron were prepared and yielded the data in Figures 15, 16, and 17. The specimens were intentionally tested in the salt-bath-annealed condition to simulate production material, so hydrogen contamination is a factor in the results. Recrystallization was found to be retarded by the presence of the contaminants when they were present in excess of 1000 ppm; probably, this also was a factor in the tensile data which would particularly affect the magnitudes of the ultimate tensile strength and elongation.

A study was made by Drolet and Martel¹⁸ on the effect of residual impurities on the ductility of alpha uranium in anticipation of the possible use of uranium in shaped charges. Two types of uranium, Grade 1 with 1000-ppm total impurities and Grade 2 with 1600 ppm were used. Grade 1 had initially been cast and Grade 2 extruded. Slices of both, 10 mm (0.4 in) thick, were vacuum annealed for 18 hours at 815°C, beta treated for 15 minutes at 700°C in a salt bath, water quenched, annealed 15 minutes in salt at 650°C, and quenched in brine. The slices were upset 50% at 380°C; tensile and hardness specimens were annealed for 18 hours in vacuum at 350, 400, 450, 500, 550, and 600°C; and then they were tested at room temperature. Figures 18 and 19 present the ductility and hardness data; included for comparative purposes are data from Taplin and Martin¹⁹ on purer material containing 460 ppm impurities. In general, hardness varies directly and ductility varies inversely with impurity content. The presence of impurities tended to refine the grain size; but, as the figures show, this was not sufficient, in itself, to promote ductility.

Figure 13. THE EFFECT OF TEMPERATURE AND ALLOY LEVEL ON THE STRENGTH OF BINARY URANIUM ALLOYS.

During the early 1960s, Nuclear Metals performed considerable research for Du Pont^(e) at Savannah River on uranium alloys for power reactors. Much of this was in connection with the alloy, uranium-350 ppm iron-900 ppm aluminum. However, some work was done on the effect of small alloy additions of other elements as well. The evaluation of a dilute alloy containing 500-ppm total of iron, nickel, silicon, and zirconium showed (by hardness tests at 200 to 500°C) that it was susceptible to heat treatment.²⁰ Also, precipitation and

(e) E. I. du Pont de Nemours and Company, Savannah River Operations, Aiken, S.C.

Figure 14. THE EFFECT OF TEMPERATURE AND ALLOY LEVEL ON THE DUCTILITY OF BINARY URANIUM ALLOYS.

Figure 15. ROOM-TEMPERATURE ULTIMATE STRENGTHS.

Figure 16. ROOM-TEMPERATURE YIELD STRENGTHS. (At 0.2% offset.)

Figure 17. ROOM-TEMPERATURE ELONGATION.

Figure 18. ROOM-TEMPERATURE TENSILE ELONGATION AND REDUCTION IN AREA OF URANIUM ANNEALED AT VARIOUS TEMPERATURES.

solid-solution hardening were found to effect an increase in uranium hardness at iron and silicon contents of about 100 ppm.²¹ Iron, silicon and/or aluminum of 100- to 150-ppm levels were shown to increase the hardness of beta-quenched uranium by as much as 50% at 200 to 600°C; however, annealing at 600°C completely erased this effect.²² In a stability test of U-0.08 wt % Si alloy, beta-quenched, it was shown that room-temperature hardness was not sensitive to aging at 600°C; but the elevated-temperature hardnesses were markedly

reduced within 8 hours.²² The improvement in mechanical properties of uranium by the addition of silicon was further verified by a French report.²³

HEAT TREATMENT

The addition of even very small concentrations of certain alloy or impurity elements to uranium changes its behavior, in response to heating and cooling through the phase transformations, as compared with unalloyed uranium. The grain size may be refined, the alpha and beta phases may become more stable at lower temperatures, recrystallization may be slowed, solid-solution and or precipitation hardening may take place, and both room-temperature and elevated-temperature properties may be changed. The extent to which any of these occurs is a function of the alloying element, its concentration, and the working and heat-treatment conditions.

Figure 19. HARDNESS OF URANIUM ANNEALED AT VARIOUS TEMPERATURES.

Microstructural Changes Resulting from Heat Treatment

Heat treatment of uranium reactor fuel alloys to increase stability under irradiation was generally accomplished by quenching from the gamma or beta phase followed by reheating or isothermally transforming in the high-alpha region. These procedures were designed both to randomize the crystal structure²⁴ and to enhance the elevated-temperature strength of the uranium, but not necessarily to optimize room-temperature properties. Early studies²⁵ showed that the fine precipitate, formed in ingot uranium containing small alloying additions of iron and silicon, grew or dissolved depending on the thermal treatment. Changes in the size and number of particles also caused changes in the beta-treated grain size. Later, more sophisticated work attempted to explain the microstructural changes which occurred and the interrelationship of various alloy additions.

Network substructures in uranium were studied by workers at Savannah River Laboratories.²⁶ These networks in unalloyed uranium were produced by the eutectoid decomposition of the gamma and beta phases. One network, corresponding to beta grain and subgrain boundaries, was composed of either incipient precipitates (visible by transmission electron microscope), when the cooling rate was rapid through the transformation, or of real particles, when the cooling rate was slow or the alloy content high. A second network, which contained uranium carbide particles, corresponded to the gamma grain boundaries and was apparent because beta nucleated at the gamma boundaries and, during cooling, either incipient or real precipitates outlined each of the beta regions as they grew into the original gamma grains. The presence of these network structures

increased the work-hardening exponent and, except in the case of high silicon, embrittled the uranium. In alloys nominally containing 30- to 500-ppm carbon and alloy additions up to 350-ppm iron, 800-ppm silicon, and 800-ppm aluminum either singly or in combination, the network appearance did not change; but intermetallics were also observed with the uranium carbide after a slow cool or an alpha anneal. Some random intermetallic precipitation also occurred. The strengths of all alloys were increased by the networks if the particles were incipient precipitates; real particles tended not to increase the strengths. Silicon probably did not contribute significantly to the network because the peritectoid decomposition of the gamma phase tends to form randomly dispersed particles.

National Lead²⁷ also contributed to an understanding of the metallurgy of dilute uranium alloys containing aluminum and iron. Two alloys (1) - 800-ppm aluminum, and (2) - 900-ppm aluminum, 400-ppm iron, 500-ppm carbon were treated at 900 to 950°C for 1 hour, transformed isothermally at 750°C for times as long as 30 hours, and water quenched. The $\gamma \rightarrow \beta + \text{UAl}_2$ eutectoidal decomposition of the first alloy was very sluggish with the substructure increasing through 16 hours at temperature. Matrix precipitation was also noticeable at 16 hours with both network and matrix particles growing through 30 hours. The addition of iron increased the rate of nucleation and appeared to increase the growth rate of the eutectoid particles. Network precipitation began in 1 minute and matrix precipitation in 2 minutes. At 30 minutes, precipitation was 90% complete, and coarsening had begun.

Effect on Alloys of Varying Heat-Treating Parameters

Alloys developed in France for reactor fuel applications were given the name Sicral which was obviously derived from the principal elements which they contained. Various modifications of this alloy system were developed. A French report on Sicral FI²⁸ (300-ppm iron, 700-ppm aluminum, 50-ppm silicon, 10-ppm chromium) contains information on the effects of variation of heat-treating and alloying parameters which should provide guidelines for a wide variety of these high uranium alloys. First, it was reported that a rapid quench from the casting temperature promoted the formation of many carbide nuclei yielding a fine as-cast grain size. Secondly, the time and temperature of the heating for the solution treatment was investigated for 31-mm-diameter X 70-mm-long alloy rods quenched from a salt bath into 20°C water. Grain size was shown to increase with decreasing quench rate, and the alpha grain size decreased with increasing time at the solution temperature but only until the UAl_2 network was completely dissolved which was accomplished in from 8 minutes at 760°C to 2 minutes at 840°C. The U_6Fe particles dissolved more rapidly than did the UAl_2 . The experiment was run at two carbon levels, 250 and 500 ppm; there was no effect of carbon on alpha grain size. It was concluded, therefore, that when the alloying elements are completely dissolved, the alpha grain size becomes independent of the time and temperature in the gamma phase before quenching, that is, independent of the gamma grain size before quenching.

Thirdly, quenching rates of from 5 to 1000°C/s were investigated. The $\beta \rightarrow \alpha$ transformation could proceed by three mechanisms as the transformation temperature was decreased from

660 to 5°C: (1) eutectoid decomposition, (2) bainitic, and (3) martensitic transformation. Maximum alpha grain refining occurred during the bainitic transformation (a situation confirmed by a Japanese investigator)²⁹ at quenching rates of 100 to 250°C/s. When the purity of an alloy was such that the bainite reaction did not occur, the capability for grain refinement was lost.

Fourthly, experiments were run to modify the alloy such that the alpha grain size might be refined in spite of the less drastic cooling and lower quenching rate possible in heavier sections. Comparisons were made using Sicral F1 with the iron removed and with the aluminum removed. The iron was found to have a greater effect on the hysteresis of the phase transformation and on the lowering of the $\beta \rightarrow \alpha$ martensite-transformation temperature than the aluminum and allowed this temperature to be attained using a lower quenching rate. However, iron was not as effective alone as in combination with aluminum. With a uranium alloy containing 200-ppm iron, 150-ppm silicon, and 500-ppm carbon, the martensitic reaction was found to take place below 500°C as compared to 430°C for Sicral F1; and the alpha grain size obtained was slightly larger. The bainitic zone was narrower; but the range of cooling rates was wider, 60 to 580°C/s with the higher being the more effective.

An alloy of 260-ppm iron and 280-ppm silicon worked well for heavier sections giving good grain refinement at lower quenching rates. All attempts to improve the quenchableness of thicker sections (by varying the iron and the aluminum failed; but with the iron-silicon alloy, quenching rates of 50 to 400°C/s yielded a 20- to 30- μ m grain size.

The rate of quenching these alloys from the solution temperature also has been shown to affect their texture.³⁰ Preferred orientation has been developed in alloys as a result of stresses arising from a number of sources during quenching. Among these are thermal gradients, volume changes resulting from phase transformations, precipitation of alloy phases, varying diffusion rates of the alloy constituents, and martensitic transformation as opposed to diffusion-controlled transformation.

The degree of texture formed depended both on cooling rate and transformation temperature. At high-cooling rates, transformation took place by a martensitic transformation at lower temperatures; and, as the cooling rate decreased, higher temperature diffusion-controlled transformation occurred. There was an initial tendency toward randomness at high-quenching rates which was followed at lower rates by maximum preferential orientation, after which a random state was again approached. The preferred orientation was such that there was a deficiency of (020) planes in sections parallel to the quenched surface.

Effect of Heat Treatment on Properties

Because of the application of these high uranium alloys in the elevated-temperature high-radiation environment of reactors, there was little interest in room-temperature mechanical properties but considerably more in hot hardness which, as previously mentioned, was believed (although probably erroneously) to be a measure of creep resistance.

Nuclear Metals performed one such study for Savannah River Laboratories³¹ using three alloys: (1) uranium-150 ppm iron-100 ppm silicon, (2) uranium-250 ppm iron-350 ppm silicon, and (3) uranium-250 ppm iron-250 ppm silicon-250 ppm aluminum. The Fe-Si alloys were beta solution treated at 730°C, but the Fe-Si-Al alloy required a gamma treatment at 800°C. Cooling was by oil quenching. Precipitation at 300 to 500°C increased the hot hardness 200% over high-purity uranium, and the second alloy became harder than the first. The third alloy was harder after the 800°C solutioning and aging than after aging following solutioning at 730°C. All the alloys softened after long times at aging temperature. Carbon in the range of 30 to 500 ppm had no effect on hot hardness.

Additional work was also done by Nuclear Metals on other alloys, uranium-350 ppm silicon, uranium-350 ppm silicon-800 ppm aluminum, and uranium-350 ppm silicon-350 ppm iron-800 ppm aluminum.³² The same general comments as above apply to the solution treatments after similar precipitation treatments (up to 100 hours at 400, 500, or 600°C), the second alloy was 5, 50, and 75% harder at room temperature, 400 and 600°C, than the first alloy. The hot hardness of alloys beta-solution-treated increased with increasing silicon and iron; gamma-solution-treated alloys showed increasing hardness with increasing silicon, iron, and aluminum. In work at National Lead,³³ room-temperature diamond-pyramid hardnesses made with loads of 5 to 3000 g showed no difference in the hardness of the uranium matrices of annealed uranium-350 ppm silicon-350 ppm iron-800 ppm aluminum alloy and high purity uranium.

Effects of Alloy Additions on Recrystallization

In the section on **Mechanical Properties**, an effect of impurities of retarding recrystallization was noted.¹⁷ This was more extensively investigated by workers at National Lead of Ohio.³⁴ Rods for fuel elements of uranium-350 ppm iron-350 ppm silicon-800 ppm aluminum were rolled and subjected to annealing studies. Annealing for 1 hour at 550°C produced little change in the as-rolled structure; and 1 hour at 600 to 660°C caused varying amounts of recrystallization, which was not complete until the time at 660°C exceeded 1 hour.

Time-Temperature-Transformation Characteristics

For pure and very low alloy uranium there has been considerable questioning as to whether the beta-to-alpha transformation proceeded by a diffusion or a shear (martensitic) process, or both. A survey of early literature³⁵ indicated that the former occurred upon cooling slowly through the phase transformation and the latter during rapid cooling. However, with higher alloying content, the transformation became more sluggish.¹

A substantial amount of work on the time-temperature-transformation (T-T-T) characteristics of several alloys was done at Mallinckrodt so that an understanding of the effects of various alloy additions was developed.³⁶ The samples used in this study were only 0.8 to 1.6 mm (1/32 to 1/16 in.) thick, so the T-T-T data developed is not applicable to heavy sections. In addition, protective coatings and oxide were found also to slow heat

conduction during the quenching. Four alloys were used in the study: (1) Alloy M, uranium-800 ppm silicon; (2) Alloy DD7, uranium-650 ppm aluminum; (3) Alloy F87 (Sicral), uranium-250 ppm iron-330 ppm silicon-670 ppm aluminum-200 ppm chromium; and (4) Alloy CS (Sicral), uranium-250 ppm iron-330 ppm silicon-930 ppm aluminum-190 ppm chromium. The first two had been gamma extruded, the third alpha extruded, and the fourth had been centrifugally cast. Data were obtained by the use of a quenching dilatometer. Figures 20 through 23 are the beta-to-alpha T-T-T curves obtained. Figures 20, 21, and 22 show that Alloys M, DD7, and F87 possessed two modes of transformation as

Figure 20. THE T-T-T DIAGRAM FOR ISOTHERMAL BETA-TO-ALPHA TRANSFORMATION OF ALLOY M (800 ppm SILICON). (The solution temperature was 732°C; the dashed lines are indicative of the uncertainty as to the absolute location and shape of the lines.)

Figure 21. THE T-T-T DIAGRAMS FOR ISOTHERMAL BETA-TO-ALPHA TRANSFORMATION OF ALLOY DD7 (650 ppm ALUMINUM). (The solution temperature was 732°C; only the upper portion of the diagram could be determined.)

indicated by the two "noses" on the curves; the extreme rapidity of transformation of Alloy DD7 precluded determination of the lower part. A difference in the transformation kinetics of Alloy CS is revealed by the three "noses" in Figure 23. The effect of quenching Alloy CS from 800°C in the gamma phase is shown in Figure 24. As compared with Figure 23, the behavior at the same alpha temperatures was different. Specimens which had been

Figure 22. THE T-T-T DIAGRAM FOR ISOTHERMAL BETA-TO-ALPHA TRANSFORMATION OF ALLOY F87 (250 ppm IRON, 330 ppm SILICON, 670 ppm ALUMINUM, 200 ppm CHROMIUM, 600 ppm CARBON). (The solution temperature was 732°C; dashed portions of the lines indicate uncertainty as to proper position.)

Figure 23. THE T-T-T DIAGRAM FOR ISOTHERMAL BETA-TO-ALPHA TRANSFORMATION OF ALLOY CS (CENTRIFUGALLY CAST, 260 ppm IRON, 330 ppm SILICON, 930 ppm ALUMINUM, 190 ppm CHROMIUM). (Solution temperature was 732°C; dashed portions of lines indicate uncertainty as to absolute position.)

Figure 24. THE T-T-T DIAGRAM FOR ISOTHERMAL TRANSFORMATION OF ALLOY CS (CENTRIFUGALLY CAST, 260 ppm IRON, 330 ppm SILICON, 930 ppm ALUMINUM, 190 ppm CHROMIUM, 60 ppm CARBON). (Quenched from the gamma phase at 800°C; dashed lines refer to behavior during initial quenches of the samples in question; solid lines represent transformation behavior in subsequent quenches.)

previously transformed and were being retransformed began their transformation later than specimens being transformed for the first time. This effect was not observed for this alloy on beta quenching as Figure 23 reveals. The delay in the start of transformation in Figure 24 as compared with Figure 23 was the consequence of increased solubility of the aluminum in the gamma phase as compared with the beta phase.

This study revealed the operation of three types of transformation kinetics. For Alloys M, DD7, and F87, down to the nose of the first "C," the transformation process was said to be diffusion-controlled nucleation and growth. From this point to the intersection with the lower "C" for these three alloys and for the upper two "Cs" for beta-quenched Alloy CS, the kinetics are similar to the bainitic transformation in steel for which the mechanism is a mixture of shear and diffusion. The lower "C" has been found to represent a time-dependent shear or martensitic-type mechanism. In general, grain size appeared to be minimized by transformation at the nose of the upper "C."

Figures 20, 23, and 24 show the relative influence of aluminum on the transformation kinetics of uranium alloys. Additional work on the influence of a few hundred parts per million of aluminum and/or iron has also been done by Haberlin and Gittus.³⁷ High iron content increased the tendency for high cooling rates to depress the $\beta \rightarrow \alpha$ transformation; it also reduced the minimum grain size and lowered the rate of the $\beta \rightarrow \alpha$ transformation for a given rate of cooling. In line with what was previously reported, aluminum had little effect

except to produce fine precipitates whose morphology was little influenced by the $\beta \rightarrow \alpha$ transformation.

DEFORMATION

Information on the deformation of uranium with respect to the influence of impurity content has been found to be limited to a small amount of data on separating forces during rolling. However, because of the value of strain hardening as a method of increasing the strength of uranium, a brief review of deformation processes and the effects of temperature on them will be included.

Hockett et al.³⁸ subjected normal purity uranium to compression tests between 20 and 300 K and found it to be a strongly work-hardening material. However, when tested above the working temperature, it was found to be unexpectedly weak; but the study did suggest that large strain deformations at warm temperatures should lead to a high yield stress at room temperature. With its high rate of strain hardening, it was predicted that a saturation flow stress of 2900 MPa (420 ksi) was attainable at room temperature. Workers at AWRE^(f) did evaluations on the effects of warm-working on uranium properties.³⁹ Uranium plates were warm-rolled up to a 90% reduction at 250°C. Tensile tests at ambient temperatures showed a progressive increase in fracture stress and ductility with increased deformation. Tensile ductility was also improved by a vacuum recrystallization anneal, and good cupping behavior was characteristic of the high-tensile-ductility material. Earlier work at AWRE⁴⁰ showed the effect of warm-rolling on the ductile-brittle transition temperature of alpha uranium. Reactor-grade uranium was hot-rolled 80% at 640°C, vacuum annealed at 600°C, and given various amounts of warm-cross-rolling. Tensile tests showed that by increasing the reduction from 10 to 40%, the transition temperature was lowered by about 60°C.

The effect of impurities on the deformation of uranium has been demonstrated by the increase in specific pressures during the rolling of uranium with various levels of impurities. A Japanese reference⁴¹ reported the effects on commercially pure uranium and uranium containing 1000 ppm carbon. At 300°C, with 12 and 19% reductions per pass, the specific-rolling pressures were 40 and 52 kg/mm² for commercially pure and 59 and 61 kg/mm² for the 1000-ppm material. On rolling at 600°C and 33% reduction per pass, the mean-rolling pressure was about 20 kg/mm² for both. Qualitative data were also obtained for rolling uranium rods of various purities.⁴² Roll-separating forces during high-alpha rolling were 10% higher for uranium-500 ppm carbon-110 ppm silicon-150 ppm iron than for unalloyed uranium (450 ppm carbon-110 ppm iron-50 ppm silicon) and were 55% higher for uranium-615 ppm carbon-365 ppm iron-330 ppm silicon-810 ppm aluminum.

(f) Atomic Weapons Research Establishment, Aldermaston, England.

REFERENCES

1. John A. Fellows, *Seven Years of Uranium Alloy Development at Weldon Spring 1959/1966*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1510 (1966).
2. J. A. Straatman and N. F. Neumann, *Equilibrium Structures in the High Uranium-Iron Alloy System*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1487 (1964).
3. R. B. Russell, *The Uranium-Iron-Aluminum Constitution Diagram - Summary Report*, Nuclear Metals Division of Textron, Inc., West Concord, Massachusetts, NMI-2813 (1964).
4. R. G. Schierding and L. A. Fergason, *Phase Studies of the Uranium-Iron-Silicon Ternary System with the Electron Microprobe*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1502 (1966).
5. N. Swindells, "The Solubility of Iron in Solid Uranium Between 0.003 Weight Percent and 0.03 Weight Percent Iron," *Journal of Nuclear Materials*, **18**, 261-271 (1966).
6. S. F. Paris, *Summary Technical Report for the Period October 1, 1964 to December 31, 1964*, R. Burgett, Ed., National Lead Company of Ohio, Cincinnati, Ohio, NLCO-940 (1965).
7. T. A. Balaeva, T. A. Alekseevko, and L. N. Aleksandrova, "Solubility of Aluminum, Silicon, and Iron in α -Uranium," "Stroenie Svoistva Splavov Dlya Atomnoi Energetiki," O. S. Ivanov, Ed., 34-38 *Izdatel'stvo Nauka*, Moscow (1973) [English transl, *Nuclear Science Abstracts*, **29**(9), 21503 (1974)].
8. J. A. Straatman and N. F. Neumann, *Equilibrium Structures in the High Uranium-Silicon System*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1486 (1964).
9. L. A. Fergason, D. L. Parker, and N. F. Neumann, "Equilibrium Phase Compositions of the Ternary Alloy U-Si-Fe by Electron Microprobe Analysis," *Process Development Quarterly Progress Report*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1484 (1964).
10. R. B. Russell, *The Uranium-Iron-Aluminum Constitution Diagram, Summary Report*, Nuclear Metals Division of Textron, Inc., West Concord, Massachusetts, NMI-2813 (1964).
11. R. G. Schierding and L. A. Fergason, *Phase Studies of the Uranium-Iron-Aluminum Ternary System with the Electron Microprobe*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1503 (1966).

12. J. A. Straatman and N. F. Neumann, *Equilibrium Structures in the High Uranium-Aluminum Alloy System*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1488 (1964).
13. L. A. Ferguson and N. F. Neumann, "Determination of the Phase Diagram for the System U-Al-Fe with an Electron Microprobe," *Process Development Quarterly Progress Report*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1482 (1963).
14. H. Inouye and A. C. Schaffhauser, *Low Temperature Ductility and Hydrogen Embrittlement--A Literature Review*, Oak Ridge National Laboratory, Oak Ridge, Tennessee, ORNL-TM-2563 (1969).
15. R. J. Wakelin, *The Influence of Impurities on the Properties of Uranium*, UKAEA Atomic Weapons Research Establishment, Aldermaston, England, AWRE O 63/70 (1970).
16. *Summary Technical Report (on Feed Material Production) for the Period April 1 - June 30, 1967*, R. Burgett, Ed., National Lead Company of Ohio, Cincinnati, Ohio, NLCO-1001 (1967).
17. R. B. Burditt et al., *The Effect of Low Levels of Contaminants on the Mechanical Properties and Weldability of Uranium*, Union Carbide Corporation, Nuclear Division, Oak Ridge Y-12 Plant, Oak Ridge, Tennessee, Y-1411 (1961).
18. J. P. Drolet and Y. Martel, *Effect of Residual Impurities on the Ductility of α -Uranium*, Defence Research Establishment, Valcartier, Courcelletle, Quebec, Canada (1974).
19. D.M.R. Taplin and J. W. Martin, "The Effect of Annealing Temperature on the Ductility and Fracture of Uranium," *Journal of Less Common Metals*, **6**, 430-433 (1964).
20. S. Isserow et al., *Power Reactor Program Progress Report to SROO USAEC for the Period May 1, 1962 to May 31, 1962*, Nuclear Metals, Inc., Concord, Massachusetts, NMI-7251 (1962).
21. S. Isserow, R. W. Anderson, and W. L. Larson, *Power Reactor Program Progress Report to SROO USAEC for the Period September 1, 1962 to September 30, 1962*, Nuclear Metals, Inc., Concord, Massachusetts, NMI-7255 (1962).
22. S. Isserow, R. W. Anderson, and W. J. Richmond, *Power Reactor Program Progress Report to SROO, USAEC for the Period January 1, 1962 to January 31, 1962*, Nuclear Metals, Inc., Concord, Massachusetts, NMI-7250 (1962).

23. J. Laniessé, H. Aubert, and P. LeBret, "Mechanical Properties of Low Concentration Uranium Alloys," Commissariat à l'Energie Atomique, Saclay, France, *Mem Sci. Rev. Met.*, **61**, 761-767 (1964), *Nuclear Science Abstract* **7940**, 19(5).
24. W. R. McDonnell and E. F. Sturcken, *Development of High Performance Uranium Metal Fuels for Savannah River Reactors*, E. I. du Pont de Nemours and Company, Savannah River Laboratories, Aiken, South Carolina, DP-MS-72-74 (1973).
25. *Process Development Quarterly Report, April-June, 1961*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1465 (1961).
26. C. L. Angerman and R. T. Huntoon, "Network Substructures in Uranium and Its Alloys," TID-21760, E. I. du Pont de Nemours and Company, Aiken, South Carolina, *Journal Less Common Metals*, **9**, 338-353 (1965).
27. W. A. Althaus and M. M. Cook, *A Contribution to the Study of Uranium-Base Dilute Aluminum Alloys*, National Lead Company of Ohio, Cincinnati, Ohio, NLCO-978 (1966).
28. P. Magnier, *Alpha Grain Refining and Metallurgical Investigation of the Uranium Alloy Sicral F1 for Fuel Elements*, Saclay Nuclear Research Center, France, CEA-R-3905 (1969) (transl. by Sandia National Laboratories, Albuquerque, New Mexico, SC-T-722464).
29. Ko Soeno, "On the β to α Transformation and Grain Refinement of Uranium and Its Dilute Alloys," *Nippon Genshiryoku Gakkaishi*, **5**, 754-759 (1963), *Nuclear Science Abstracts*, **17**(24), Abstract 41468.
30. K. R. Riggs and N. F. Neumann, *Effect of Alloy Additions on the Quench Induced Texture of Alpha-Uranium*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1491 (1965).
31. R. W. Anderson, *Effect of Heat Treatment on the Properties of High Uranium Alloys*, Nuclear Metals, Inc., West Concord, Massachusetts, NMI-7020-2 (1964).
32. R. W. Anderson, *Effect of Heat Treatment on the Properties of High Uranium Alloys Containing Additions of Iron, Silicon, and Aluminum*, Nuclear Metals Division of Textron, Inc., West Concord, Massachusetts, NMI-7097-1 (1964).
33. E. A. Gerlach, "Microhardness of a Uranium-Base Alloy Containing 350 ppm Fe, 350 ppm Si, and 800 ppm Al (Alloy 801) Compared with High Purity Uranium," *Summary Technical Report for the Period July 1, 1968 to September 30, 1968*, National Lead Company of Ohio, Cincinnati, Ohio, NLCO-1025.

34. R. S. Haeckl, *Annealing Study of a Heavily Worked Uranium-Base Alloy Containing 350 ppm Fe, 350 ppm Si, and 800 ppm Al (Alloy 801)*, National Lead Company of Ohio, Cincinnati, Ohio, NLCO-1025 (1968).
35. J. Burke and P. H. Dixon, *The $\alpha \rightleftharpoons \beta$ Phase Transformation in Pure Uranium*, *Journal Nuclear Material*, **7**(1), 38-45 (1962).
36. N. F. Nolan, *The Beta-to-Alpha Transformation Characteristics of Some Dilute Uranium Alloys*, Mallinckrodt Chemical Works, Uranium Division, Weldon Spring, Missouri, MCW-1506 (1966).
37. "Fuel and Fertile Materials," *Reactor Materials*, **8**(4), 175-95 (Winter 1965-66). M. M. Haberlin and J. H. Gittus, *Continuous Transformation Characteristics of U-Fe-Al Alloys*, British Report TRG-590 (1964).
38. J. E. Hockett, P. S. Gilman, and O. D. Sherby, "Comprehensive Deformation of Polycrystalline Uranium at Low Temperature," *Journal Nuclear Material*, **64**(3), 231-240 (1977).
39. A. N. Hughes, R. A. Lane, and S. Orman, "Effects of Warm Work on Some Mechanical and Fabrication Properties of Uranium," *Journal Nuclear Material*, **48**(2), 172-182 (1973).
40. J. Bolton and P. D. Tilbury, "The Influence of Warm Rolling Upon the Ductile/Brittle Transition Temperature of α -Uranium," *Journal of the Institute of Metals*, **92**(3), 95 (1963).
41. Hitoshi, Watanabi, "Ductility of Metallic Uranium Containing Carbide Inclusions at Rolling," *Nippon Genshiryoku Gakkaishi*, **8**, 70-75 (1966). *Nuclear Science Abstracts*, **20**, (10), 17001.
42. H. W. Hesselbrock, "The Effect of Alloy Content on the Rolling Properties of Uranium Rods," *Summary Technical Report for the Period April 1, 1964 to June 30, 1964*, R. Burgett, Ed., National Lead Company of Ohio, Cincinnati, Ohio, NLCO-920 (1964).

Distribution

Department of Energy - Oak Ridge

Hickman, H. D.
Leed, R. E.
Poteat, R. M.

Lawrence Livermore National Laboratory

Arnold, W. F.
Bish, W. R.
Copeland, A. B.
Woelffer, R. A.
Wood, D. H.

Los Alamos National Scientific Laboratory

Hoyt, H. C.

Oak Ridge Gaseous Diffusion Plant

Armstrong, R. C.
Stief, S. S.
Wilcox, W. J., Jr.

Oak Ridge National Laboratory

Hopkins, C. C.

Oak Ridge Y-12 Plant

Anderson, R. C.
Batch, J. E./Lockett, S. W.
Beck, D. E.
Bieber, C. R.
Burditt, R. B.
Chilcoat, T. R./Smith, J. G.
Dillon, J. J.
Dodson, W. H./Googin, J. M.
Evans, P. A.
Fraser, R. J.
Jessen, N. C., Jr.
Keith, A.
Kite, H. T.
Koger, J. W.
Kollie, T. G.
Ludwig, R. L. (5)
McElroy, B. D.
Mills, J. M., Jr.
Montgomery, C. D.

Morrow, M. K.
Northcutt, W. G., Jr.
Thompson, J. E.
Townsend, A. B.
Y-12 Central Files (master copy)
Y-12 Central Files (route copy)
Y-12 Central Files (Y-12RC)
Y-12 Central Files (5)

Paducah Gaseous Diffusion Plant

Bewley, H. D.

Rockwell International - Rocky Flats

Jackson, R. J.

Sandia National Laboratories - Albuquerque

Eckelmeyer, K. H.

Sandia National Laboratories - Livermore

Adolphson, D. R.
Hoover, W. R.
Mote, M. W., Jr.
Spencer, W. J.

Union Carbide Corporation - New York

Tinsley, S. W.

In addition, this report is distributed in accordance with the Category UC-25, **Materials**, as given in the *Standard Distribution for Unclassified Scientific and Technical Reports*, DOE/TIC-4500.

4--2213

ABSTRACT

A survey of the literature has been made on the experimental results of small additions of iron, silicon, and aluminum to uranium. Information is also included on the constitution, mechanical properties, heat treatment, and deformation of various binary and ternary alloys.

24 figs, 13 tables, 42 references,

Date of Issue: **December 31, 1980**
Distribution Category: **UC-25**

Y-2213

**LOW ALLOY ADDITIONS OF IRON, SILICON, AND
ALUMINUM TO URANIUM — A LITERATURE SURVEY**

R. L. Ludwig
Metals and Ceramics Department
Y-12 Development Division

Oak Ridge Y-12 Plant

P. O. Box Y, Oak Ridge, Tennessee 37830

Prepared for the Department of Energy
Under US Government Contract W-7405-eng-26