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Columbus, Ohio

GRAIN REFINEMENT OF URANIUM BY ALLOYING

Contract No. W-7405-eng-92

Prepared by:

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SUMMARY

Binary alloys of uranium with 38 elements in the range 0.01 to 1.0 atomic per cent were made. Three alloys having nominal compositions of 0.01, 0.1, and 1.0 atomic per cent were made with most of the elements, and in addition, 12 ternary and quaternary alloys were made. The alloys were cast, rolled to 7/8-inch-diameter bars, heat treated, and examined for grain size. A section of each "as rolled" bar was sent to Hanford Laboratory for additional examination.

After a 600°C. anneal, binary alloys with 16 of the elements had failed to recrystallize completely, and 9 of the ternary and quaternary alloys also had cold-worked or partially recrystallized structures. Ten elements, either singly or in combination, caused a marked grain refinement in beta-quenched metal. Chromium was the most effective of these elements. Six other elements were somewhat less effective in reducing the grain size of beta-quenched uranium.

After annealing at 800°C., only 5 alloys had grain sizes less than 0.20 mm. Alloys of Ni, Sn, Au, Pd, and Th contained a precipitate at the grain boundary of a relatively fine grain structure, but also had a coarse grain structure superimposed on this network of precipitate. Alloys with Pb, W, Co, Fe, B, Al, Sb, and Mo also contained a second phase in the form of randomly distributed particles.

EXPERIMENTAL WORK

Melting

The alloys were prepared by melting in an induction furnace under vacuum or an argon atmosphere. Figure 1 shows the 50-kw., 300-kc. Megatherm power source and the general arrangement of the furnace. The arrangement of heater, crucible, and mold inside of the 6-inch quartz tube and pouring chamber is shown in Figure 2.

All of the alloys were melted in graphite crucibles and cast into a 1-7/8-inch I.D. graphite mold. A new crucible was required for each heat, but the mold and stopper could be reused for several heats.

The stopper was a zirconia ball supported by a molybdenum rod. This stopper was withdrawn by a spring when a solenoid-operated locking pin was released.

The vacuum-pumping system consisted of a 15-c.f.m. fore pump and a 6-inch oil-diffusion pump. The furnace was pumped down to approximately 5 microns pressure before the power was turned on. As the temperature increased, however, the pressure tended to increase because the capacity of the pumps was not large enough to keep up with the gas evolved. When the pressure reached about 50 microns, arcing occurred inside the furnace tube, and it was necessary to turn off the power until the pressure was reduced below this critical point.

It was found that an argon atmosphere during the melt-down prevented oxidation and also eliminated the above arcing. Consequently, most of the later heats were made under one atmosphere pressure of argon. The furnace was pumped down to 5 microns pressure and flushed with argon.

FIGURE 1. VACUUM MELTING FURNACE

65863

090 005

FIGURE 2. SCHEMATIC DRAWING OF MELTING FURNACE

O-18696
77047

090 006

A static pressure slightly above atmospheric was maintained in order to prevent air from entering the system.

Three methods of introducing the alloying elements were used. Most of the additions were made by adding the element with the uranium charge or by adding a master alloy of the element to the charge. Those elements having high vapor pressures, such as zinc, cadmium, and calcium, were placed in the mold and the uranium poured on top of them.

The master alloys, containing 5 to 10 weight per cent of the alloying element, were made in a small induction furnace in beryllia crucibles. They were cooled in the crucible and analyzed at the top and bottom to detect segregation.

Table I shows the nominal composition of the alloys and the conditions under which they were made. Binary alloys with 38 elements were made, as well as 12 ternary or quaternary alloys. Three unalloyed heats also were made for use as standards.

Three 9-pound heats, having nominal compositions of 0.01, 0.1, and 1.0 atomic per cent, were made with most of the elements. Alloys with higher percentages of Al, Be, Ti, B, Cr, and Si also were made. In addition to these binary alloys, ternary and quaternary alloys were made with some of those elements which were most effective as grain refiners in the binary alloys.

The chemical analyses of the alloys also are shown in Table I. These analyses were made on drillings taken 1 to 2 inches from each end of the bars after rolling to 7/8-inch diameter. The analyses show that there was considerable segregation in the alloys. Apparently, little or no inductive stirring occurred because of the high frequency of the power source, and little mixing resulted from bottom pouring. All of the samples to be heat treated were taken adjacent to the drillings at the top end of

TABLE I. ANALYSIS AND GRAIN SIZE OF HEAT-TREATED ALLOYS

Alloy No.	Nominal Composition, Atomic %	Analysis (Atomic %)		Casting Conditions			Average Grain Diameter, mm.			
		Top Of Casting	Bottom Of Casting	Furnace Atmosphere	Pouring Temperature, °C.	Method of Addition	As Rolled	Heated 1 Hour At 600°C., Furnace Cooled	Heated 5 min. At 700°C., Water Quenched	Heated 1 Hour At 800°C., Furnace Cooled
HU1	No addition	-	-	Vacuum	1220	No addition	0.045	0.065	0.150	>0.2
HU2	No addition	-	-	"	1205	No addition	0.065	0.065	0.200	>0.2
HU3	No addition	2.0 C	1.4 C	"	1210	No addition	0.045	0.045	0.120	>0.2
HFe1	1.0 Fe	1.7 Fe; 2.52 C	0.66 Fe; 2.0 C	"	1304	Direct	Cold worked ⁽¹⁾	Cold worked ⁽¹⁾	0.065 ^(1,2)	>0.2 ⁽¹⁾
HFe2	0.1 Fe	0.17 Fe; 2.0 C	0.15 Fe; 2.0 C	"	1210	Direct	0.045	0.035	0.120	>0.2
HFe3	0.01 Fe	0.09 Fe; 2.71 C	0.08 Fe; 2.0 C	"	1182	Direct	0.065	0.045	0.090	>0.2
HN11	1.0 Ni	0.92 Ni; 1.56 C	0.44 Ni; 1.37 C	"	1288	Direct	0.065	0.045	0.150	>0.2 ⁽¹⁾
HN12	0.1 Ni	0.08 Ni; 2.0 C	0.08 Ni; 2.0 C	"	1204	Direct	0.045	0.045	0.150	>0.2
HN13	0.01 Ni	0.024 Ni; 2.0 C	0.024 Ni; 1.56 C	"	1216	Direct	0.065	0.065	0.200	>0.2
HMn1	1.0 Mn	1.34 Mn; 3.26 C	0.55 Mn; 1.37 C	"	1204	Direct	Cold worked	Cold worked	0.025 ⁽³⁾	>0.2
HMn2	0.1 Mn	0.1 Mn; 2.33 C	0.095 Mn; 1.56 C	"	1232	Direct	0.045	0.065	0.065	>0.2
HMn3	0.01 Mn	0.009 Mn; 2.89 C	0.013 Mn; 1.56 C	"	1204	Direct	0.065	0.065	0.150	>0.2
HSn1	1.0 Sn	0.32 Sn	0.08 Sn	"	1232	Direct	Cold worked	Cold worked ⁽⁴⁾	0.150	>0.2 ⁽¹⁾
HSn2	0.1 Sn	0.002 Sn	0.006 Sn	"	1232	Master alloy	0.045	0.045	0.120	>0.2
HSn3	0.01 Sn	0.014 Sn	0.004 Sn	"	1232	Master alloy	0.045	0.065	0.120	>0.2
HCu1	1.0 Cu	8.7 Cu	0.54 Cu	"	1232	Direct	0.045	0.045	0.090	>0.2
HCu2	0.1 Cu	0.19 Cu	0.18 Cu	"	1260	Master alloy	0.045	0.065	0.090	>0.2
HCu3	0.01 Cu	0.033 Cu	0.011 Cu	"	1232	Master alloy	0.065	0.065	0.120	>0.2
HAu1	1.0 Au	0.93 Au; 1.56 C	0.88 Au; 1.56 C	"	1216	Direct	Cold worked	Cold worked	0.120	>0.2 ⁽¹⁾
HAu2	0.1 Au	0.054 Au; 1.56 C	0.012 Au; 1.56 C	"	1216	Direct	0.045	0.065	0.120	>0.2
HAu3	0.01 Au	0.012 Au; 1.95 C	0.066 Au; 1.37 C	"	1232	Direct	0.065	0.065	0.120	>0.2
HIIn1	1.0 In	0.041 In	0.031 In	"	1260	Direct	0.065	0.065	0.120	>0.2
HIIn2	0.1 In	<0.02 In	<0.02 In	"	1232	Direct	0.065	0.065	0.120	>0.2
HIIn3	0.01 In	0.02 In	0.031 In	"	1204	Direct	0.045	0.045	0.20	>0.2
HT11	1.0 Ti	0.05 Ti	0.05 Ti	"	1288	Master alloy	Cold worked ⁽⁴⁾	Cold worked ⁽⁴⁾	0.120	>0.2
HT12	0.1 Ti	0.25 Ti	0.020 Ti	"	1216	Master alloy	0.065	0.065	0.120	>0.2
HT13	0.01 Ti	<0.005 Ti	0.005 Ti	"	1216	Master alloy	0.045	0.065	0.120	>0.2
HT14	5.0 Ti	0.75 Ti	0.60 Ti	"	1232	Master alloy	Cold worked	Cold worked ⁽⁴⁾	0.120	>0.2
HT15	10.0 Ti	2.80 Ti	2.30 Ti	"	1260	Direct	Cold worked	Cold worked ⁽⁴⁾	0.065	>0.2
HAg1	1.0 Ag	16.7 Ag	<0.044 Ag	"	1232	Direct	Cold worked	Cold worked ⁽⁴⁾	0.120	>0.2
HAg2	0.1 Ag	<0.044 Ag	<0.044 Ag	"	1204	Direct	0.035	0.045	0.150	>0.2

TABLE I. ANALYSIS AND GRAIN SIZE OF HEAT-TREATED ALLOYS (CONTINUED)

Alloy No.	Nominal Composition, Atomic %	Analysis (Atomic %)		Casting Conditions			Average Grain Diameter, mm.			
		Top Of Casting	Bottom Of Casting	Furnace Atmosphere	Pouring Temperature, °C.	Method of Addition	As Rolled	Heated 1 hour at 600°C., Furnace Cooled	Heated 5 min. at 700°C., Water Quenched	Heated 1 hour at 800°C., Furnace Cooled
HAg3	0.01 Ag	<0.044 Ag	<0.044 Ag	Vacuum	1260	Direct	0.065	0.045	0.120	>0.2
HPd1	1.0 Pd	1.23 Pd	0.63 Pd	"	1216	Direct	Cold worked	Cold worked	0.120	>0.2(1)
HPd2	0.1 Pd	0.15 Pd	0.14 Pd	"	1232	Direct	0.035	0.045	0.120	>0.2(1)
HPd3	0.01 Pd	0.065 Pd	0.050 Pd	"	1232	Direct	0.065	0.045	0.150	>0.2
HBe1	1.0 Be	<0.13 Be; 1.56 C	0.53 Be; 1.56 C	"	1232	Master alloy	0.045	0.065	0.120	>0.2
HBe2	0.1 Be	<0.013 Be	<0.013 Be	"	1260	Master alloy	0.045	0.065	0.150	>0.2
HBe4	4.0 Be	0.53 Be	0.26 Be	"	—	Master alloy	0.045	0.065	0.090	>0.2
HA11	1.0 Al	0.26 Al	0.09 Al	"	1204	Master alloy	0.065	0.065	0.150	>0.2
HA12	0.1 Al	0.18 Al	0.04 Al	"	1204	Direct	0.065	0.045	0.120	>0.2
HA13	0.01 Al	0.35 Al	0.04 Al	"	1204	Direct	0.045	0.045	0.120	>0.2
HA14	4.0 Al	3.53 Al	3.00 Al	"	—	Direct	Cold worked	Cold worked	0.150(1)	>0.2(1)
HS11	1.0 Si	0.93 Si	0.93 Si	"	1232	Master alloy	Cold worked	0.065	0.065(1,2)	>0.120(1)
HS12	0.1 Si	0.051 Si	0.094 Si	"	1232	Master alloy	0.035	0.035	0.090(2)	>0.150
HS13	0.01 Si	0.042 Si	0.094 Si	"	1316	Master alloy	0.065	0.045	0.120	>0.2
HCo1	1.0 Co	4.1 Co	1.70 Co	"	—	Master alloy	Cold worked(1)	Cold worked(1)	0.150-0.2(1,2)	>0.2(1)
HCo2	0.1 Co	0.32 Co	0.20 Co	"	1204	Master alloy	Cold worked	Cold worked(4)	0.120	>0.2
HCo3	0.01 Co	0.08 Co	0.04 Co	"	1232	Master alloy	0.045	0.045	0.20	>0.2
HTh1	1.0 Th	0.36 Th; 2.0 C	0.072 Th; 1.56 C	"	1249	Direct	0.035	0.035	0.120(1)	>0.2(1)
HTh2	0.1 Th	<0.031 Th	<0.031 Th	"	1232	Master alloy	0.045	0.065	0.090	>0.2
HTh3	0.01 Th	<0.031 Th	0.031 Th	"	1204	Master alloy	0.065	0.065(4,1)	0.150	>0.2
HSb1	1.0 Sb	0.39 Sb	0.36 Sb	"	1232	Master alloy	Cold worked(4,1)	0.065(4,1)	0.120	>0.2(1)
HSb2	0.1 Sb	<0.04 Sb	<0.04 Sb	"	1204	Master alloy	Cold worked	0.015(4)	0.150	>0.2
HSb3	0.01 Sb	<0.04 Sb	0.04 Sb	"	1232	Master alloy	0.065	0.065	0.150	>0.2
HZr1	1.0 Zr	0.63 Zr	0.73 Zr	"	—	Master alloy	0.065(4)	0.065(4)	0.065	>0.2
HZr2	0.1 Zr	0.08 Zr	0.08 Zr	"	1204	Master alloy	0.045	0.065	0.150	>0.2
HZr3	0.01 Zr	0.05 Zr	0.03 Zr	"	1316	Master alloy	0.065(4,3)	0.015(4)	0.090	>0.2
HW1	1.0 W	0.70 W	0.47 W	"	1204	Master alloy	Cold worked(1)	Cold worked(1)	0.120(1)	>0.20(1)
HW2	0.1 W	0.065 W	0.052 W	"	1232	Master alloy	0.045	0.045	0.120	>0.2
HW3	0.01 W	<0.013 W	<0.013 W	"	1260	Master alloy	0.065	0.065	0.150	>0.2
HCB1	1.0 Cb	0.078 Cb	0.078 Cb	"	1204	Master alloy	0.035	0.035	0.120	>0.2
HCB2	0.1 Cb	0.078 Cb	0.078 Cb	"	1204	Master alloy	0.065	0.065	0.150	>0.2
HCB3	0.01 Cb	0.078 Cb	0.013 Cb	"	1204	Master alloy	0.045	0.065	0.150	>0.2
HCr1	1.0 Cr	1.24 Cr; 1.56 C	1.15 Cr; 1.56 C	"	1204	Master alloy	Cold worked	Cold worked(4)	0.035(3)	>0.150(3)
HCr2	0.1 Cr	0.183 Cr	0.151 Cr	"	1204	Master alloy	Cold worked	Cold worked	0.045	>0.2
HCr3	0.01 Cr	0.023 Cr	0.023 Cr	"	1232	Master alloy	0.065	0.065	0.120	>0.2
HPt1	1.0 Pt	1.32 Pt	1.35 Pt	"	1232	Master alloy	Cold worked	0.045(2)	0.120(2)	>0.090(2)
HPt2	0.1 Pt	0.064 Pt	0.076 Pt	"	1232	Master alloy	0.045	0.045	0.120	>0.2
HPt3	0.01 Pt	0.025 Pt	0.013 Pt	"	1204	Master alloy	0.045	0.065	0.150	>0.2
HB11	1.0 Bi	0.03 Bi; 1.56 C	0.03 Bi; 1.18 C	"	1232	Master alloy	0.045(2)	0.065	0.150	>0.2
HB12	0.1 Bi	<0.012 Bi	<0.012 Bi	"	1204	Master alloy	0.065	0.065	0.150	>0.2
HB13	0.01 Bi	<0.006 Bi	<0.006 Bi	"	1204	Master alloy	Cold worked(2)	0.035	0.065	>0.2

TABLE I. ANALYSIS AND GRAIN SIZE OF HEAT-TREATED ALLOYS (CONTINUED)

Alloy No.	Nominal Composition, Atomic %	Analysis (Atomic %)		Casting Conditions			Average Grain Diameter, mm.			
		Top Of Casting	Bottom Of Casting	Furnace Atmosphere	Pouring Temperature, °C.	Method of Addition	As Rolled	Heated 1 Hour at 600°C., Furnace Cooled	Heated 5 min. at 700°C., Water Quenched	Heated 1 Hour at 800°C., Furnace Cooled
HCe1	1.0 Ce	0.102 Ce; 5.3 C	0.051 Ce; 2.35 C	Argon	1260	Master alloy	0.090	0.120	0.150	>0.2
HCe2	0.1 Ce	<0.017 Ce	<0.017 Ce	Argon	1260	Master alloy	0.20	0.120	0.150	>0.2
HCe3	0.01 Ce	<0.017 Ce	<0.017 Ce	Argon	1232	Master alloy	0.035	0.065	0.120	>0.2
HTa1	1.0 Ta	0.344 Ta	0.423 Ta	Vacuum	1304	Master alloy	0.045	0.045	0.120	>0.2
HTa2	0.1 Ta	0.066 Ta	0.066 Ta	Vacuum	1232	Master alloy	0.045	0.045	0.120	>0.2
HTa3	0.01 Ta	0.066 Ta	0.013 Ta	Vacuum	1204	Master alloy	0.045	0.035	0.150	>0.2
HV2	0.1 V	0.14 V	0.14 V	Vacuum	1204	Master alloy	0.035	0.035	0.120	0.090
HV3	0.01 V	0.09 V	0.023 V	Vacuum	1232	Master alloy	0.045	0.045	0.120	>0.2
HV4	0.5 V	0.61 V	0.37 V	Vacuum	1204	Master alloy	0.035	0.035	0.120	>0.2
HFeV1	0.5 V; 0.5 Fe	0.61 V; 0.60 Fe	0.37 V; 0.38 Fe	Vacuum	1132	Master alloy	Cold worked	0.045	0.045	>0.2
HPb1	1.0 Pb	0.86 Pb	0.78 Pb	Argon	1260	Master alloy	0.045 ⁽¹⁾	0.045 ⁽¹⁾	0.120 ⁽¹⁾	>0.2 ⁽¹⁾
HPb2	0.1 Pb	0.13 Pb	0.12 Pb	Argon	1260	Direct	Cold worked ⁽¹⁾	Cold worked ^(1,2)	0.120 ⁽¹⁾	>0.2 ⁽¹⁾
HPb3	0.01 Pb	0.003 Pb	0.003 Pb	Argon	1260	Direct	Cold worked ⁽¹⁾	Cold worked ⁽¹⁾	0.090 ⁽¹⁾	>0.2 ⁽¹⁾
HB1	1.0 B	0.55 B	<0.22 B	Argon	1238	Direct	0.035	0.045	0.065	>0.2 ⁽¹⁾
HB2	0.1 B	0.44 B	<0.22 B	Argon	1260	Direct	0.045	0.045	0.120	>0.2
HB4	2.5 B	0.37 B	0.26 B	Argon	1260	Direct	0.035	0.045	0.090	>0.2
HCo1	1.0 Cd	<0.001 Cd	0.028 Cd	Argon	1260	Mold	0.045	0.065	0.150	>0.2
HCo2	0.1 Cd	0.002 Cd	<0.001 Cd	Argon	1271	Mold	0.045	0.065	>0.2	>0.2
HCo3	0.01 Cd	<0.001 Cd	<0.001 Cd	Argon	1260	Mold	0.045	0.065	0.150	>0.2
HZn1	1.0 Zn	0.002 Zn	0.011 Zn	Argon	1260	Mold	0.065	0.065	0.120	>0.2
HZn2	0.1 Zn	0.003 Zn	0.006 Zn	Argon	1260	Mold	Cold worked	Cold worked ⁽²⁾	0.120	>0.2
HZn3	0.01 Zn	<0.002 Zn	<0.002 Zn	Argon	1260	Mold	0.045	0.065	0.150	>0.2
HMo1	1.0 Mo	0.97 Mo	0.94 Mo	Argon	1316	Direct	Cold worked	Cold worked	0.065	0.150
HMo2	0.1 Mo	0.14 Mo	0.132 Mo	Argon	1274	Direct	Cold worked	0.045	0.090	>0.2
HMo3	0.01 Mo	0.042 Mo	0.032 Mo	Argon	1274	Direct	Cold worked	0.045	0.120	>0.2
HGe1	1.0 Ge	0.695 Ge	0.66 Ge	Argon	1204	Master alloy	Cold worked	Cold worked	0.120	>0.2
HGe2	0.1 Ge	0.36 Ge	0.33 Ge	Argon	1232	Master alloy	0.045	0.035	0.150	>0.2
HGe3	0.01 Ge	0.16 Ge	0.16 Ge	Vacuum	—	Master alloy	0.045	0.045	0.120	>0.2
HMg1	1.0 Mg	0.05 Mg	0.34 Mg	Vacuum	1316	Mold	0.045	0.065	0.20	>0.2
HMg3	0.01 Mg	0.15 Mg	0.02 Mg	Vacuum	1343	Mold	0.065	0.065	0.2	>0.2
HCa1	1.0 Ca	0.18 Ca	0.06 Ca	Vacuum	1316	Mold	0.065	0.065	0.2	>0.2
HLi1	1.0 Li	<0.033 Li	<0.03 Li	Vacuum	1260	Mold	0.090	0.090	>0.2	>0.2
HTe1	1.0 Te	0.43 Te	0.056 Te	Vacuum	1260	Mold	0.045	0.045	>0.2	>0.2
HBa1	0.5 Ba	0.087 Ba	<0.035 Ba	Vacuum	1260	Direct	0.035	0.065	0.090	>0.2
HSr1	0.5 Sr	<0.13 Sr	<0.13 Sr	Vacuum	1260	Direct	0.035	0.035	0.090	>0.2
HGa1	1.0 Ga	2.04 Ga	1.26 Ga	Vacuum	1260	Direct	Cold worked	Cold worked ⁽²⁾	0.090	>0.2
H1	1.0 Zr; 0.1 Cr	1.23 Zr; 0.091 Cr; 2.89 C	0.37 Zr; 0.147 Cr; 2.14 C	Argon	1316	Master alloy	Cold worked	Cold worked ⁽²⁾	0.035	0.20
H2	1.0 Zr; 1.0 Si; 0.1 Cr	0.288 Zr; 0.85 Si; 0.25 Cr; 2.33 C	0.018 Zr; 0.51 Si; 0.091 Cr; 1.95 C	Argon	1260	Master alloy	Cold worked	Cold worked	0.090	0.120
H3	0.01 Zr; 0.5 Si; 0.1 Cr	0.003 Zr; 0.85 Si; 0.21 Cr	<0.001 Zr; 0.17 Si; 0.04 Cr	Argon	1260	Master alloy	Cold worked	Cold worked	0.065	0.20

TABLE I. ANALYSIS AND GRAIN SIZE OF HEAT-TREATED ALLOYS (CONTINUED)

Alloy No.	Nominal Composition, Atomic %	Analysis (Atomic %)		Casting Conditions			As Rolled	Average Grain Diameter, mm.		
		Top Of Casting	Bottom Of Casting	Furnace Atmosphere	Pouring Temperature, °C.	Method of Addition		Heated 1 Hour at 600°C., Furnace Cooled	Heated 5 min. at 700°C., Water Quenched	Heated 1 Hour at 800°C., Furnace Cooled
H4	1.0 Zr; 1.0 Si	0.14 Zr; 0.34 Si	0.02 Zr; 0.20 Si	Argon	1260	Master alloy	Cold worked	0.045	0.065	>0.2
H5	0.01 Zr; 1.0 Si	0.003 Zr; 1.53 Si	0.003 Zr; 0.43 Si	Argon	1260	Master alloy	Cold worked	Cold worked ⁽¹⁾	0.090-0.120	0.20
H6	0.5 Si	1.45 Si	0.34 Si	Argon	1288	Master alloy	Cold worked	Cold worked ⁽²⁾	0.065-0.090	0.20
H7	3.0 Si	—	—	Argon	1316	Master alloy	—	—	—	—
H8	0.5 Zr	0.55 Zr	0.21 Zr	Argon	1316	Master alloy	0.045	0.045	0.065-0.090	>0.2
H9	1.0 Zr	0.42 Zr	0.21 Zr	Argon	1316	Master alloy	0.035	0.045	0.120	>0.2
H11	2.0 Cr	0.60 Cr; 2.33 C	0.41 Cr; 2.51 C	Argon	1316	Master alloy	Cold worked	0.045	0.045	>0.2
H12	0.1 Cr	0.78 Cr	0.46 Cr	Argon	1316	Master alloy	Cold worked	Cold worked ⁽²⁾	0.015	>0.2
H13	0.3 Cr	0.91 Cr	0.09 Cr	Argon	1316	Master alloy	Cold worked	0.045	0.045	>0.2
H14	0.6 Cr	0.73 Cr	0.18 Cr	Argon	1316	Master alloy	Cold worked	Cold worked ⁽²⁾	0.045	>0.2
H15	0.01 Zr	0.001 Zr	0.001 Zr	Argon	1316	Master alloy	0.045	0.065	0.120	>0.2
H16	0.05 Zr	0.008 Zr	0.021 Zr	Argon	1316	Master alloy	0.045	0.045	0.120	>0.2
H17	0.1 Zr	0.42 Zr	0.003 Zr	Argon	1316	Master alloy	0.035	0.035	0.120	>0.2
H18	0.005 Zr	0.002 Zr	0.001 Zr	Argon	1316	Master alloy	0.035	0.045	0.120	>0.2
H19	2.0 Zr	0.36 Zr	0.003 Zr	Argon	1316	Master alloy	0.035	0.045	0.065-0.090	>0.2
H20	0.03 Zr; 0.1 Cr	0.015 Zr; 0.08 Cr	0.004 Zr; 0.10 Cr	Argon	1316	Master alloy	0.025	0.045	0.065-0.090	>0.2
H21	0.005 Zr; 0.1 Cr	<0.001 Zr; 0.14 Cr	0.001 Zr; 0.08 Cr	Argon	1316	Master alloy	0.025	0.045	0.090-0.120	>0.2
H22	0.03 Zr; 0.3 Cr	0.003 Zr; 0.82 Cr	0.001 Zr; 0.14 Cr	Argon	1316	Master alloy	Cold worked	0.025	0.065	>0.2
H23	1.5 Si	1.62 Si	1.53 Si	Argon	1316	Direct	Cold worked ⁽¹⁾	0.035 ⁽¹⁾	0.065 ⁽¹⁾	>0.2 ⁽¹⁾
H24	0.2 Si	0.21 Si	0.21 Si	Argon	—	Master alloy	0.035	0.035	0.090	>0.2
H25	0.3 Si	0.33 Si	0.29 Si	Argon	—	Master alloy	0.045	0.045	0.20	>0.2
H26	0.4 Si	0.46 Si	0.44 Si	Argon	—	Master alloy	Cold worked	0.015	0.065-0.090	>0.2
H28	0.5 Si; 0.01 Cr	0.59 Si; 0.03 Cr	0.60 Si; 0.04 Cr	Argon	1482	Master alloy	Cold worked	Cold worked	0.120	>0.2
H29	1.0 Si; 0.1 Cr	1.70 Si; 0.12 Cr	0.83 Si; 0.10 Cr	Argon	1593	Master alloy	Cold worked	Cold worked	No grains	>0.2
H30	0.5 Si; 0.01 Zr	0.94 Si; 0.21 Zr	0.59 Si; <0.001 Zr	Argon	1538	Master alloy	0.035	0.065	0.120	>0.2

- (1) Two phase
 (2) Polarized light
 (3) Segregated
 (4) Partially recrystallized

the bars; consequently, the "top" analysis probably represents fairly well the composition of these samples.

Some pick-up of carbon from the crucible occurred. The uranium used for all of the heats was alpha-rolled bar stock which contained about 0.05 weight per cent carbon. After melting, the carbon content of the alloys averaged 0.10 weight per cent. The effect of this increase in carbon content on the grain size of otherwise unalloyed uranium was negligible. Samples of the original bar stock and of the unalloyed casting HUI had the same grain size after each of the three heat treatments used.

Fabrication

The 1-7/8-inch-diameter castings were rolled at 600°C. to 7/8-inch-diameter bars. This reduction was made in seven passes, and the bars were reheated after each pass. All of the alloys rolled satisfactorily at this temperature except Alloy H7 containing 3.0% silicon. This alloy cracked in the first pass and could not be rolled further. In view of this result, the 1.5% silicon alloy, H23, was rolled at 800°C. There was a tendency for the alloys to fin in the first pass, and as a result of this, some seams were present in the finished bars. In general, however, the quality of the rolled bars was good.

In rolling the bars, the bottom of the casting was made the leading end of the bar so that chemical analyses at each end of the finished bar could be related to the top and bottom of the casting. A section 1 to 2 inches long was cut from each of the rolled bars and the ends of the bar were drilled then for chemical analysis.

A section, roughly 12 inches long, was cut from the leading end (bottom of the casting) and sent to Hanford Metallurgical Laboratory for further study. Four 3/4-inch-long sections were cut from the trailing end of the bar for examination of the as-rolled structure and for heat treatment.

Heat Treatment and Examination

Three heat treatments were given to the 3/4-inch sections of each bar. One section was heated for 1 hour at 600°C. in a vacuum and furnace cooled. The second section was heated 5 minutes in a lead bath at 700°C. and quenched in ice water. The last sample was annealed 1 hour at 800°C. in a vacuum and furnace cooled.

These heat-treated samples were split with a submerged cut-off wheel, and the longitudinal cross section of each was electrolytically polished and etched in a chromic-acetic acid bath. The grain size was determined by visually comparing the bright-field image of the sample on the ground-glass screen of a Metallograph with the ASTM standard grain-size chart for non-ferrous metals.⁽¹⁾ A few of the alloys, particularly the Pt, Fe, Co, and Si alloys, did not etch satisfactorily and were examined under polarized light after electropolishing in the chromic-acetic acid bath.

The grain sizes of the alpha-annealed and the gamma-annealed samples were easily estimated, because the grains were generally fairly uniform in size and appeared equiaxed. The transformed structure resulting from the beta quench, however, consisted of irregular-shaped grains which varied widely in size. The average grain size of this

(1) ASTM, Volume 29, Part 1, Plate VII.

structure was difficult to estimate.

Figures 3, 4, and 5 show typical alpha-annealed, beta-quenched, and gamma-annealed structures. Table I contains the results of the examination for grain size.

DISCUSSION OF RESULTS

As Rolled

In the "as-rolled" condition, the three heats having no alloy additions had typical alpha-annealed structures and an average grain size of 0.045 to 0.065 mm. The addition of Fe, Mn, Sn, Au, Ti, Pd, Si, Co, W, Cr, Ge, Mo, Sb, Pt, Al, and Ga resulted in partially recrystallized or cold-worked structures. In most of the alloys, only the nominal 1% alloy had not recrystallized, but smaller additions of Co, Cr, Sb, and Mo also prevented recrystallization during rolling. The effect of lead was unusual in that the nominal 1% lead alloy had recrystallized during rolling, but the two alloys with nominal 0.1 and 0.01 per cent lead were cold worked.

The high-silver alloy, HAg1, also had a cold-worked structure. This alloy was severely segregated, and there were indications that the

C90 014

RECEIVED

75X

Chromic-Acetic Etch

59905

FIGURE 3. TYPICAL STRUCTURE OF ALPHA-ANNEALED URANIUM

75X

Chromic-Acetic Etch

59724

FIGURE 4. TYPICAL STRUCTURE OF BETA-QUENCHED URANIUM

DECLASSIFIED

090 015

75X

Chromic-Acetic Etch

59746

FIGURE 5. TYPICAL STRUCTURE OF GAMMA-ANNEALED URANIUM

75X

Chromic-Acetic Etch

59909

FIGURE 6. STRUCTURE OF ALPHA-ANNEALED CERIUM
ALLOY, HCe2

melt had separated into two immiscible liquids. The "as-rolled" sample, however, did not contain any of the silver-rich phase.

Two other alloys, HBi3 and HZn2, had failed to recrystallize during rolling. These alloys contained 0.005% Bi and 0.003% Zn, respectively. It is doubtful whether the addition of these small quantities of Bi and Zn caused this change in the recrystallization characteristics, because other heats with similar quantities of Bi and Zn were unaffected by the addition. No explanation for the unusual behavior of these two alloys was found.

Cerium had a unique effect on the as-rolled structure. The structure of the nominal 1.0 and 0.1% cerium alloys was similar to that found in beta-quenched uranium. The grains were irregular in shape and were much larger than those found in the standards.

Alpha Annealed

Annealing 1 hour at 600°C. and furnace cooling caused no significant change in the grain size of those alloys which had recrystallized during hot rolling. Seven of the alloys with cold-worked structures had recrystallized, but the remainder of these alloys were unaffected by the alpha anneal. Additions of Fe, Mn, Sn, Au, Ti, Ag, Pd, Al, Co, W, Cr, Si, Pb, Zn, Mo, Ge, and Ga apparently raised the recrystallization temperature or decreased the rate of recrystallization so that these alloys remained in the cold-worked condition.

With most of the above elements, only the nominal 1% alloys had not recrystallized, although 0.1% Co and Cr alloys had also failed to recrystallize. The structure of the Pb and Zn alloys was not changed by this alpha anneal, but the low-Bi alloy, HBi3, had recrystallized. One binary Si alloy, No. H6, had failed to recrystallize, but this alloy was

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090 017

badly segregated. Other Si alloys which were more uniform in composition had recrystallized.

Six of the ternary or quaternary alloys did not recrystallize completely during this alpha anneal. All of these alloys contained one or more of the elements which affected the recrystallization of the binary alloys.

The structure of the nominal 1.0 and 0.1% Ce alloys was unchanged by annealing at 600°C. Figure 6 shows the structure of the 0.1% Ce alloy, HCe2, after annealing. As in the rolled sample, the grain size was larger than that of any other alpha-annealed alloy, and the structure resembled that of beta-quenched uranium.

Beta Quench

Because a beta heat treatment will randomize the orientation of alpha-rolled uranium,⁽¹⁾ a beta-quenched alloy with an average grain diameter approaching that of alpha-rolled and annealed uranium, 0.065 mm. or less, would fulfill the requirements for fine-grained, randomly oriented metal. Therefore, the grain size of the beta-quenched samples was considered the most significant of any of the heat-treated samples.

Table I shows the grain size of the unalloyed standards and the alloys after heating 5 minutes at 700°C. and quenching in ice water. The unalloyed uranium had an average grain diameter of 0.120 to 0.20 mm., and the grains had the typical irregular shape and numerous twins or slip lines, as shown in Figure 4.

The majority of the alloys had structures and grain sizes similar to those of the standards. Binary additions of Fe, Mn, Ti, Zr, Cr, Si, Bi, B, Mo, and Ga, however, resulted in an average grain diameter of 0.065 mm. or less. With the exception of Cr and Bi, only the binary alloys containing

(1)ANL 4150, p. 16.

a nominal 1.0% or more of the above elements had this fine a grain size. Small additions of Fe, Mn, Si, Zr, B, and Mo, as well as additions of Cu, Be, Th, Pb, Ba, and Sr, resulted in the slightly larger grain size of 0.090 mm.

In addition to the above binary alloys which had an average grain diameter of 0.065 mm. or less, four ternary alloys, HFeV1, H1, H4, and H22, which contained combinations of Fe, V, Zr, Si, and Cr, also had grain sizes in this range. One other ternary alloy, H20, had a slightly larger grain size and one, H29, containing Si and Cr could not be etched to show the grain size. Several etchants were tried on this alloy without success and examination under polarized light also failed to show the grain structure. This alloy, however, had numerous cracks resulting from the quench and probably would not be useful for this reason.

Two quaternary alloys with Zr, Si, and Cr, Alloys H2 and H3, had grain sizes of 0.065 and 0.090 respectively.

The quantity of addition required to effect grain refinement in the ternary and quaternary alloys appeared to be about the same as in the binary alloys, and no significant refinement in grain size resulted from the addition of the third or fourth element.

Those alloys which showed a refinement in grain size after heating 5 minutes at 700°C. and quenching were investigated further. Samples of the rolled bar of each alloy were heated 15 minutes at 700°C. in a lead pot and quenched in ice water. Table II shows the results of this additional heat treatment, as well as the results of the initial test.

After the longer soaking time at 700°C., only the binary alloys of Mn, Si, Cr, B, and Mo had an average grain diameter of 0.065 mm. or less. All of those ternary and quaternary alloys which had a refined grain size after initial heat treatment also were fine grained after the 15-minute

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TABLE II. ALLOYS SHOWING GRAIN REFINEMENT
AFTER A BETA QUENCH

Alloy No.	Analysis, Atomic %		Average Grain Diameter, mm.	
	Top	Bottom	5 minutes at 700°C. and Quenched	15 minutes at 700°C. and Quenched
HU1	--	--	0.150	0.150
HU2	--	--	0.200	0.200
HU3	2.0 C	1.4 C	0.120	0.150
HFe1	1.7 Fe	0.66 Fe	0.065	0.090
HFe3	0.09 Fe	0.08 Fe	0.090	0.150
HMn1	1.34 Mn	0.55 Mn	0.065	0.045
HMn2	0.10 Mn	0.095 Mn	0.090	0.120
HCu1	8.7 Cu	0.54 Cu	0.090	0.090
HCu2	0.19 Cu	0.18 Cu	0.090	0.120
HT15	2.8 Ti	2.3 Ti	0.065	0.090
HBe4	0.53 Be	0.26 Be	0.090	0.20
HSi1	0.93 Si	0.93 Si	0.065	0.065
HSi2	0.05 Si	0.09 Si	0.090	0.150
H6	1.45 Si	0.34 Si	0.065-0.090	0.090
H24	0.21 Si	0.21 Si	0.090	0.120
H26	0.46 Si	0.44 Si	0.065-0.090	0.065
HTh2	0.03 Th	0.03 Th	0.090	0.150
HZr1	0.63 Zr	0.73 Zr	0.065	0.090
HZr3	0.05 Zr	0.03 Zr	0.090	0.120
H8	0.55 Zr	0.21 Zr	0.065-0.090	0.120
HL9	0.36 Zr	0.003 Zr	0.065-0.090	0.150
HCr1	1.24 Cr	1.15 Cr	0.035	0.065
HCr2	0.18 Cr	0.15 Cr	0.065	0.065-0.090
HL1	0.60 Cr	0.41 Cr	0.045	0.045
HL2	0.78 Cr	0.46 Cr	0.015	0.035
HL3	0.91 Cr	0.09 Cr	0.065	0.065-0.090
HL4	0.73 Cr	0.18 Cr	0.065	0.045
HBI3	0.006 Bi	0.006 Bi	0.065	0.120
HPb3	0.003 Pb	0.003 Pb	0.090	0.150
HB1	0.55 B	0.22 B	0.065	0.065
HB4	0.37 B	0.26 B	0.090	0.090
HMo1	0.97 Mo	0.94 Mo	0.065	0.065
HMo2	0.14 Mo	0.13 Mo	0.090	0.120
HBa1	0.87 Ba	0.035 Ba	0.090	0.150
HSr1	0.13 Sr	0.13 Sr	0.090	0.120
HGa1	2.04 Ga	1.26 Ga	0.065	0.065
HFeV1	0.60 Fe; 0.61 V	0.38 Fe; 0.37 V	0.045	0.065
H1	1.23 Zr; 0.09 Cr	0.37 Zr; 0.15 Cr	0.035	0.045
H2	0.29 Zr; 0.85 Si; 0.25 Cr	0.02 Zr; 0.51 Si; 0.09 Cr	0.90	0.065

TABLE II. ALLOYS SHOWING GRAIN REFINEMENT
AFTER A BETA QUENCH (CONTINUED)

Alloy No.	Analysis, Atomic %		Average Grain Diameter, mm.	
	Top	Bottom	5 minutes at 700°C. and Quenched	15 minutes at 700°C. and Quenched
H3	0.003 Zr; 0.85 Si; 0.21 Cr	0.001 Zr; 0.04 Cr; 0.17 Si	0.065	0.045-0.065
H4	0.14 Zr; 0.34 Si	0.02 Zr; 0.20 Si	0.065	0.045-0.065
H20	0.015 Zr; 0.08 Cr	0.004 Zr; 0.10 Cr	0.065-0.090	0.090
H22	0.003 Zr; 0.82 Cr	0.001 Zr; 0.14 Cr	0.065	0.065
H29	1.70 Si; 0.12 Cr	0.83 Si; 0.10 Cr	— *	—

* Etch failed to show grain structure.

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080 021

beta soak and quench. Binary alloys of Fe, Cu, Ti, Ga, and Zr had slightly refined grain sizes, but these additions were not so effective as the elements indicated above. The alloys of Be, Th, Bi, Ba, and Sr which had shown a refined grain size after the initial heat treatment were coarse grained after the longer soak in the beta region.

Chromium was the most effective grain-refining addition of all of the elements tried. Figure 7 shows the effect of increasing amounts of chromium on the grain size after heating 5 minutes at 700°C. and quenching. An addition of 0.18 atomic per cent, Alloy HCr2, resulted in an average grain diameter of 0.045 mm., and larger amounts further decreased the grain size.

Figures 8 and 9 show the effects of silicon and manganese on grain size after the initial beta quench. Alloys HSi 2 and HSi3, containing 0.05 and 0.042% silicon, respectively, had grain sizes similar to that of the unalloyed standards. Alloy H24, containing 0.21% silicon had a slightly refined grain size after the 5-minute beta heat treatment, but was coarse grained after the 15-minute soak and quench. Alloys H26 and HSi1, containing 0.46 and 0.93% silicon, respectively, were fine grained after both heat treatments. Increasing the silicon content to 1.62%, as in Alloy H23, did not further reduce the grain size obtained under these conditions.

Manganese was also an effective grain-refining agent. Alloy HMn2, containing 0.10% manganese, had an average grain diameter of 0.065 mm. and the higher manganese alloys, HMn1, had an average grain diameter of 0.035 mm. The latter alloy was severely segregated, and only the outer portion of the rolled bar had this refined grain size. The center of the bar was coarse grained.

75X

Chromic Etch
Alloy HCr3, 0.023 Per Cent Cr

70485

75X

Chromic Etch
Alloy HCr2, 0.18 Per Cent Cr

73785

75X

Chromic Etch
Alloy H12, 0.78 Per Cent Cr

70480

75X

Chromic Etch
Alloy HCr1, 1.24 Per Cent Cr

73781

FIGURE 7. THE EFFECT OF CHROMIUM ON THE STRUCTURE OF
BETA-QUENCHED URANIUM

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090 023

75X

Chromic Etch

59724

Alloy HMn3, 0.009 Per Cent Mn

75X

Chromic Etch

59723

Alloy HMn2, 0.10 Per Cent Mn

75X

Chromic Etch

70115

Alloy HMn1, 1.34 Per Cent Mn

FIGURE 8. THE EFFECT OF MANGANESE ON THE STRUCTURE OF BETA-QUENCHED URANIUM

75X

73776

Picric Etch
Alloy HSi2, 0.05 Per Cent Si

75X

73779

Picric Etch
Alloy HSi1, 0.93 Per Cent Si

75X

73784

Picric Etch
Alloy H23, 1.62 Per Cent Si

FIGURE 9. THE EFFECT OF SILICON ON THE STRUCTURE
OF BETA-QUENCHED URANIUM

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Boron, molybdenum, and gallium also had a major refining effect on the beta-quenched grain size. The structure of these alloys is shown in Figure 10. Somewhat larger quantities of these elements, however, were required to produce a grain size of 0.065 mm., or less, than in the case of chromium, manganese, or silicon.

Gamma Anneal

After a gamma anneal of one hour at 800°C., followed by furnace cooling, the unalloyed standards, as well as most of the alloys, had an average grain diameter much larger than 0.200 mm. Only additions of Cr, V, Pt, Si, and Mo reduced the grain size to less than 0.20 mm., and the effect of these elements was not consistent in all cases with the quantity of the element indicated by the analyses. The nominal 0.1% V alloy had a grain size of 0.090 mm., but the alloy containing 0.37 to 0.61% V was as coarse grained as the standards. In the case of Si, both the nominal 1.0 and 0.1% alloys had a somewhat refined grain size but the grain size of Alloys H24, H25, H26, and H23, containing 0.21, 0.33, 0.46, and 1.62% of Si, respectively, were as large as that of the standards. Only the nominal 1% alloys of Cr, Pt, and Mo had refined grain sizes.

Several of the alloys contained a second phase which formed a grain-boundary network around a small primary grain structure. Superimposed over this network was the usual large grain structure resulting from the gamma anneal. Figure 11 shows this structure in the nickel and thorium alloys. Alloys with tin, palladium, gold, and iron also had similar structures.

A second phase in the form of more or less rounded particles or stringers was observed in alloys of tungsten, cobalt, lead, boron, antimony,

75X

Chromic Etch
Alloy HBI, 0.55 Per Cent B

59741

75X

Chromic Etch
Alloy HGaI, 2.04 Per Cent Ga

70113

75X

Picric Etch
Alloy HMol, 0.97 Per Cent Mo

73782

FIGURE 10. THE EFFECT OF BORON, GALLIUM, AND MOLYBDENUM
ON THE STRUCTURE OF BETA-QUENCHED URANIUM

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090 027

75X

Chromic-Acetic Etch
Alloy HTh1, 0.36 Per Cent Thorium

59735

75X

Chromic-Acetic Etch
Alloy HNi1, 0.92 Per Cent Nickel

59721

FIGURE 11. STRUCTURE OF GAMMA-ANNEALED
URANIUM ALLOYS SHOWING IN-
TERGRANULAR NETWORK

aluminum, molybdenum, and silicon. Figure 12 shows the structure of the tungsten, cobalt, and lead alloys.

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080 029

75X Chromic Etch 59734
Alloy HCoI, 4.1 Per Cent Cobalt

75X Chromic Etch 59738
Alloy HWI, 0.70 Per Cent Tungsten

75X Chromic Etch 70120
Alloy HPbI, 0.86 Per Cent Lead

FIGURE 12. STRUCTURE OF GAMMA-ANNEALED URANIUM
ALLOYS-SHOWING TWO PHASES