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THE PREPARATION AND PHYSICAL PROPERTIES OF URANIUM BINARY ALLOYS
CONTAINING ALUMINUM AND COLUMBIUM

by

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URANIUM TECHNOLOGY

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ABSTRACT

The effects of small additions of aluminum or columbium to uranium were investigated in order to find an alloy addition agent which would substantially increase the yield strength and creep resistance of uranium. The use of aluminum as an addition agent was first investigated, but early work indicated the use of aluminum to be impractical. The composition of the aluminum-uranium alloy was difficult to control, and the physical properties of the as-cast alloys were extremely erratic. The response of the aluminum alloy to solution treatment and aging was inconsistent and the structures obtained were unpredictable. Although considerable melting difficulties were encountered during the investigation of the uranium-columbium alloys, the physical properties of the as-cast alloys were found to be considerably superior to unalloyed uranium, and the yield strengths of the columbium alloys could be further elevated by heat treatment.

INTRODUCTION

Experiments were conducted to determine an alloy addition agent for uranium which would substantially increase the yield strength and creep resistance of uranium.

In addition it was required that the following specifications be met:

1. The alloy and a heat treatment, if required, were to be developed in the shortest possible time.
3. Alloying additions were to be held to a minimum and were not to exceed five atomic percent.
4. Ductility and corrosion resistance were to be maintained within reasonable limits.
5. It was necessary that the alloy be adaptable to production melting techniques.

Results of Literature Search.

Previous reports on uranium alloy systems were studied to determine the alloying constituents most likely to raise the tensile properties of uranium. From the preliminary survey uranium-aluminum alloys appeared to offer the most promise. Work reported by Gordon (1), Buzzard and Cleaves (3) indicated that the uranium rich uranium-aluminum alloys were age hardenable. The low alloy content ranges of

the uranium-columbium system were indicated by the work of Greninger (3) and Sawyer (4) to be worthy of further investigation. The literature search also indicated that binary alloys of uranium with manganese, chromium, or platinum would possibly yield elevated tensile properties. Except for one melt of 2.5 atomic percent manganese which was extremely brittle in the as-cast condition, the latter three systems were not investigated.

Outline of Investigation Procedures.

The investigations were carried out as follows:

1. The first alloy heats were cast to form three one-half inch diameter by three inch long bars. The top of one bar and the bottom of another from each melt were machined and the chips analyzed to check composition and indicate homogeneity. The bars were sectioned into one-half inch lengths for hardness determinations and photomicrographic studies of the as-cast condition and subsequent to a variety of heat treatments.
2. When hardness determinations indicated a composition or a heat treatment which yielded interesting properties, standard 0.505 inch tensile bars were cast for tensile studies.
3. When a heated and quenched bar yielded more desirable properties than as-cast bars of the same composition, Jominy end quench tests were performed to indicate the effective depth of quench.

4. The relative atmospheric corrosion resistance of the alloys was determined by observation of the machined surfaces of test bars after prolonged exposure to air.
5. As a final check on the desirability of a particular alloy or heat treatment creep tests were performed.

The uranium-aluminum system, which, at the beginning of the program, was thought to be the most promising, was investigated first. When sufficient data had been collected to indicate that the uranium-aluminum alloys were not desirable, the investigation of this system was dropped completely and the investigation of uranium-columbium alloys undertaken.

Equipment and Materials Used

Most of the alloy metals were made using Ames high purity uranium containing approximately 50 ppm of carbon. However, some metals were made with normal uranium containing 300-500 ppm of carbon. When interesting properties were disclosed in alloys made of normal uranium, similar compositions were made with high purity metal to verify the results.

Melts were made in induction heated vacuum furnaces with pressures varying between 10-1000 microns of mercury. The alloys, except for a few special melts noted later, were melted in magnesia crucibles with magnesia stopper rods and poured into alumina or magnesia lined graphite molds to minimize contamination.

High temperature heating (740°C - 1050°C) for heat treating was accomplished by supporting the piece with platinum wire in a salt bath of NaCl or a mixture of twenty weight percent NaCl, 25 percent KCl and 55 percent BaCl_2 . The salt was heated in a graphite crucible in an induction furnace. The temperature of the bath could be closely regulated by manual control of the wattage input to the induction coil. Quenching was done in a water bath with a delay between the salt bath and the quench tank of 1-5 seconds. Aging and low temperature heating (up to 600°C) were done in an induction heated vacuum annealing furnace.

Melting temperatures were determined by means of a Leeds and Northrup Optical Pyrometer, Catalog No. 8266C. The temperature readings were taken on the metal by viewing through a pyrex sight glass in the furnace top. Salt bath heat treating temperatures were determined by means of a chromel-alumel thermocouple immersed in the salt. Temperatures for the low temperature vacuum treatments were determined by a chromel-alumel thermocouple placed next to the piece within the vacuum furnace.

Tensile determinations were made on standard 0.505 inch diameter test bars using a Southwark-Emery testing machine with a maximum loading capacity of 120,000 pounds. The tensile specimens were loaded at the rate of 1200 pounds per minute. In most cases the tensile data show that the ratio of stress to strain was not consistent over a measurable range. The yield strength was determined by a 0.2

percent offset line parallel to an approximated modulus line.

Room temperature creep tests were conducted on standard 0.505 inch tensile bars statically loaded for periods varying from 300 to 1000 hours. In general, the loading time was limited to 300 hours, as previous creep test results on normal uranium bars showed that the slopes of the time-elongation plot obtained during the first 300 hours was indicative of the slope of the plot up to 1000 hours, see Fig. 1.

ALUMINUM ALLOY INVESTIGATIONS

A total of fourteen melts were made in which aluminum, varying from 0.1 to 4.0 atomic percent, was the alloying element. Although increased hardness and elevated tensile properties were obtained from these alloys in both the as-cast and heat treated conditions (as compared with unalloyed uranium), the variations in metal structure and tensile results indicated that uranium-aluminum alloys, as obtained during this program, were not suitable for production applications.

Uranium-Aluminum Alloy Melting Problems

The desired composition of aluminum in the alloy was difficult to achieve. Loss of aluminum due to volatilization during the melting caused deviations from the calculated alloy content which could not be determined readily. Experience showed that variations in melting time, temperature, or pressure gave alloys whose aluminum content could not be predicted. The necessity for precise control of these variables was a disadvantage from the practical aspect of production

STRAIN vs. TIME CURVES

Showing Relative Creep Resistance of
Uranium - Columbium Alloy and Normal Uranium Bars

Fig. 14

work. It was felt that aluminum losses could be better controlled by melting under a controlled pressure of inert gas to minimize aluminum volatilization. This was not attempted as later work disclosed the aluminum alloys to be generally undesirable.

Samples taken from the top and bottom of each casting generally showed a wide spread in aluminum content indicating incomplete homogenization, and no consistent trend of segregation in the castings was noted. To cite one case, a 2.5 atomic percent aluminum alloy was desired. The charge was made up for a 3.5 atomic percent addition of aluminum. The alloy was melted, held between 1250-1280°C for fifteen minutes at a pressure of 300-500 microns of mercury, and poured at 1280°C. A casting was obtained which analyzed 1.64 atomic percent aluminum at the top and 2.38 atomic percent aluminum at the bottom.

When melting alloys containing 2.5 atomic percent or greater amounts of aluminum, a reaction occurred after melt down. This reaction has been reported by N. P. Allen (5) for alloys containing greater than 5 weight percent (31.7 atomic percent) aluminum in uranium as occurring with considerable violence, evolution of heat, and spattering of metal. Although spattering of metal was never observed in the more dilute alloys melted in this program, the reaction caused sufficiently vigorous agitation in a number of heats, charged to yield a 2.5 atomic percent aluminum alloy, that the stopper rod was dislodged with a premature pour resulting.

Heat Treating Procedures for Uranium-Aluminum Alloy

It was felt that solution heat treatment followed by controlled precipitation of the UAl_2 phase would be required to achieve the maximum advantage of the alloy addition. In the paper by Gordon (1), a solution treatment of $2\frac{1}{2}$ atomic percent aluminum alloy at 1040°C , followed by a water quench, was reported to have retained complete solution of the UAl_2 phase. A number of bars of the low aluminum as-cast alloy were given a heat treatment which consisted of a solution treatment at 1000 or 1050°C followed by a water quench. Treatment at this temperature proved impractical from a production viewpoint for the following reasons:

1. There was considerable variance in hardness between different samples of the same analysis after identical treatments and also considerable hardness variance between adjacent indentations on the same piece.
2. Photomicrographic studies showed what appeared to be quench cracks subsequent to the treatment. For an example see Figure 2.
3. The alloy at these temperatures was semi-plastic and deformation of a cast shape could be expected to result.
4. Oxidation of the metal was high.
5. Quenching control of cast shapes would be difficult from temperatures above 1000°C .

In view of the difficulties experienced by solution treatment above 1000°C , treatment at lower temperatures within the gamma-

Figure 2: This 100X photomicrograph shows a 1.0 atomic percent aluminum alloy which had been heated to 1000°C for two hours and water quenched. The cracks shown are believed to be caused by the abrupt quench. The cracks severely affected the ductility of the alloy and further work on solution treatment of aluminum alloys at this temperature was abandoned as impractical.

uranium temperature range was investigated. Uranium-aluminum alloys containing from 0.9 to 3.6 atomic percent aluminum were solution treated at 800, 850 or 900°C for from one-quarter to four hours at temperature. The hardness values, taken across the face of each 1/2 inch diameter solution treated and quenched test slug, were very erratic. The values varied as much as 11 points Rockwell C with average deviation from the median value of 2 points Rockwell C. The averaged hardness values of each heat treated alloy piece, when plotted as hardness versus time at temperature, were also highly erratic and failed to indicate any hardness trends. Figure 3 is included as an example of the inconsistent hardnesses resulting from the solution treatment of uranium-aluminum alloys within the gamma-uranium temperature range. Because of the inconsistent hardnesses obtained by the solution heat treatment, aging of these alloys was not attempted.

The solid solubilities of aluminum in uranium are reported to be 0.82 atomic percent aluminum at 640°C in the alpha phase and 1.23 atomic percent at the beta-gamma transformation (4). These values indicated that the investigation of solution treatment within the beta-uranium range, followed by lower temperature aging in the alpha range might be worthy of investigation. Solution treatment of alloy specimens containing 0.5 to 1.5 atomic percent aluminum at 740°C for 1/2 to 4 hours at temperature, followed by a water quench, yielded slightly increased and comparatively consistent hardnesses as shown in Figure 4. Aging at 350°C subsequent to the beta range treatment failed to materially

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change the hardness of the samples tested.

General Characteristics of Uranium-Aluminum Alloys

Uranium-aluminum alloys containing more than approximately 2.5 atomic percent aluminum were very brittle in the as-cast condition. This was demonstrated when samples containing 2.9 atomic percent aluminum cracked when stamped with identifying numbers and an as-cast tensile bar containing 3.8 atomic percent aluminum tested 56,000 p.s.i. ultimate strength with no appreciable elongation or reduction in area.

Microscopic studies of the alloy samples in the as-cast and heat treated condition failed to indicate the cause of the erratic hardnesses obtained. The metallographic structures appeared somewhat complex and the same structures appeared in both the hard and soft samples. Typical uranium-aluminum alloy structures are shown in Figure 5.

The atmospheric corrosion resistance of uranium alloys containing aluminum appeared superior to unalloyed uranium. Machined bars exposed to the atmosphere did not appear to gain a dark oxide coating as rapidly as unalloyed bars.

The tensile properties of some uranium-aluminum alloys are shown in Table A. Creep resistance values were not determined, as it was felt that more desirable alloys could be obtained from other binary systems.

COLUMBIUM ALLOY INVESTIGATIONS

The columbium-uranium alloys which were properly melted and

Treatment: As-Cast
Average Hardness: 20 Rockwell C

Treatment: 850°C for one-half hour
and water quench.
Average Hardness: 37 Rockwell C

Treatment: 850°C for 1 hour and
water quench

Average Hardness: 27 Rockwell C

Figure 5: These 100X Photomicrographs show the different structures obtained by varied treatments of samples from the same casting analyzing 1.5 atomic percent aluminum. The average hardnesses taken from the polished specimens are given. The varied structures and erratic hardnesses shown indicate the difficulties encountered in reproducing hardness values with aluminum alloys.

TABLE I
TENSILE VALUES OF URANIUM-ALUMINUM ALLOYS

<u>Melt No.</u>	<u>Analysis at % Al</u>	<u>Treatment See (1)</u>	<u>Ultimate Str. psi</u>	<u>Yield Str. psi</u>	<u>Elong. % in 2"</u>	<u>R. A. %</u>
3109	1.1	As-cast	108,700	40,000	10.9	9.8
3109	1.1	a	103,750	49,000	4.7	3.2
7125	1.4	As-cast	89,400	42,500	3.1	2.8
3125	2.2	As-cast	98,000	46,500	3.1	2.8
4125	3.8	As-cast	56,600	55,000	0.4	0.4 (2)
4125	3.8	a	22,550	--	-	- (2)
4125	3.8	b	Bar fractured under initial load (2)			

(1) Heat treatments

a. 740°C for 1½ hours and water quenched

b. 1000°C for 2 hours and water quenched

(2) Brittle fractures

cast had tensile and creep resistance properties considerably superior to unalloyed uranium. These alloys had a fine grain structure with reproducible hardnesses, elevated yield strength, high ductility, and increased resistance to creep in both the as-cast and heat treated conditions. However, the alloy was difficult to prepare by the usual uranium melting and casting procedures and considerable investigative work to discover the best alloying techniques was necessary. The first uranium-columbium alloy castings were made by adding 60 mesh columbium powder to the uranium charge, heating the mixture to 1300°C for from $1/2$ to $3/4$ hour, and pouring the metal into a ceramic lined graphite bar mold. A few of the castings so produced were acceptable for investigative work, but analysis of the castings generally showed wide variances from the columbium content desired, and fracture of the alloy bars often showed shrinkage defects.

Uranium-Columbium Alloy Melting Difficulties

The inability to control the analysis of the alloy castings was shown early in the program to be due to difficulties in gaining positive solution of the columbium in the uranium. In many cases less than half of the columbium added to the charge had been taken into solution in the uranium. As is shown by the uranium-columbium phase diagram (2), the metals are infinitely soluble in both the liquid and high temperature gamma solid phases. The liquidus shows a continuous curve between the melting points of uranium (1130°C) and columbium (2415°C). On the basis of these data, it was considered advisable to

to raise the melt temperature and hold the melt for a longer time in order to gain more complete solution of the columbium. A few melts were made where the temperature was raised to as high as 1550°C and held at temperature for from 1 to 2 hours. Melts held at temperatures above 1450°C were rarely successfully poured due to stopper rod failure. Attack of the magnesia crucible, stopper rod and furnace insulation was severe at temperatures above 1450°C . Both magnesia and magnesium metal were heavily condensed on the furnace shell subsequent to a high temperature run and upon exposure to air the magnesium would flash and burn in a hazardous fashion.

A variety of crucible materials, less subject to break down under vacuum conditions than magnesia, were tried in order to elevate the alloy homogenization temperature. High temperature melts were made in small crucibles of cerium sulfide, beryllia, zirconia, and zircon. The latter two materials showed the most promise as they withstood temperatures of 1575°C for $1\frac{1}{2}$ hours without visible sign of attack. However, chemical analysis of the alloy slugs melted in the zirconia and zircon crucibles indicated a silicon (120-500 ppm respectively) and iron (40-110 ppm respectively) pickup.

Alloy melts were made in magnesia crucibles under partial pressures of helium and argon in an attempt to minimize the magnesia attack at elevated homogenizing temperatures. It was found that melting under inert gas partial pressure (2-6 inches of mercury) successfully minimized magnesia crucible attack at temperatures up to 1480°C .

However the inert gas caused conduction and convection heat losses which limited operating temperatures to 1480°C . Analysis of the castings showed that less of the columbium was dissolved during these melts under partial pressure than with comparable melts utilizing vacuum techniques. The reluctance of the columbium to dissolve was believed to be increased by the lack of outgassing agitation within melts made under partial pressures.

It was thought that the analysis of final castings might be controlled by remelting alloyed stock in which the weights of metal had been adjusted according to the analyzed columbium content to yield a casting of the desired analysis. Without exception, the analysis of the final casting was erratic due, probably, to the undissolved columbium powder trapped in the metal, the flotation of the columbium (a phenomena which will be described later), and the inability to obtain a truly representative sample.

In order to lower the temperatures required for solution of the columbium, a sintered compact of uranium and columbium powders was tried as a means of introducing the columbium to the melt. The compact contained twelve atomic percent columbium which should have a melting point of approximately 1350°C (interpolated from the phase diagram) if the compact was completely homogenized. The sintered compacts only partially dissolved in the uranium metal when held at 1400°C for one and a half hours. After the melt was poured, the master alloy compacts retained in the crucible appeared to have been differentially leached.

An attempt was made to make a master alloy of columbium and nickel in which the nickel was used as a carrier for the columbium to gain low temperature solution of columbium in uranium. The charge contained 40 atomic percent columbium and remainder nickel, which conforms with an eutectic composition melting at 1175°C (6). The eutectic contains Ni_3Cb as one of the phases and it was hoped that the presence of the intermetallic compound would alloy brittle fracture of the master alloy. Such a physical condition of the alloy would facilitate making the charge and afford a large surface area for solution in the melt. After melting the master alloy, the magnesia crucible showed severe attack beneath the metal surface. The resulting alloy was hard and did not yield to ready fracture. Work with the nickel-columbium master alloy was carried no further as it was felt that the physical characteristics were not desirable and the composition of the master alloy would be difficult to control. In addition, the use of nickel would lower the purity of the uranium alloy and could alter the properties of the alloy in an undesirable fashion.

The most satisfactory method of making a columbium alloy addition to uranium was found to be with columbium-uranium alloy buttons prepared in the reduction bomb. Alloy buttons containing 1.5, 5, and 10 atomic percent columbium in uranium were prepared by Benjamin R. Hayward and these buttons have been used as master alloy additions for both experimental and production castings. The alloy buttons were made by adding either columbium powdered metal or Cb_2O_5 to the standard

reduction charge. Reference is made to LA 472, Preparation of Uranium Metal by Stationary Bomb Method by R. D. Baker and a report now in the process of preparation by Hayward on the bomb production of columbium-uranium master alloy buttons. The master alloy in all composition ranges was readily soluble in uranium when held at 1350°C for one-half hour. The final compositions of the alloy castings could be held within ± 0.25 atomic percent when the master alloy biscuits were used.

The flotation of columbium in alloyed melts, which has been previously mentioned, was indicated by chemical analysis of a number of alloy melts. When slugs of alloy were sectioned and analyzed subsequent to freezing in the crucible the upper portions of the ingot generally contained greater amounts of columbium and carbon. Columbium concentrations at the top of the casting occurred also in large static cast shapes where the metal was poured at a sufficient temperature to cause a delay in the freezing of the metal after pouring. Centrifugally cast shapes consistently showed a higher columbium and carbon content in the center of the castings, indicating that the top of the melt or the last metal poured contained a columbium concentration. The tendency for the flotation of columbium was more pronounced in melts made with normal uranium containing 100-500 ppm. of carbon than when high purity containing 30-100 ppm of carbon was used. This phenomena was not completely investigated but sufficient data have been collected to indicate the cause. The consistent relationship between high carbon content with high columbium content and machining difficulties encountered in areas of

alloy concentration indicated the presence of hard carbides. It is believed that columbium, when added to a uranium charge containing appreciable carbon, combined to form either a columbium carbide or possibly a complex columbium-uranium carbide upon heating of the charge and tended to rise within the melt. Microscopic studies have shown a concentration of carbides in the high columbium content regions, but these carbides have the physical appearance of uranium carbide. The flotation of the columbium can best be controlled by the use of low carbon melting stock.

Uranium-Columbium Casting Difficulties

Most of the early castings produced for tensile tests of columbium-uranium alloys were defective due to shrinkage cavities within the tensile bars. The bar stock had been cast in the same manner that tensile bars for uranium had been made in the past. The metal was cast into an Alundum lined graphite mold to yield a 1 inch diameter bar, 6 inches long and headed at one end with a large diameter feeder head. In the past, no defects were noticeable in uranium tensile bars cast in this fashion, but columbium alloy bars cast in this type mold had low ductility and a shiny, spongy area was generally revealed at the tensile fracture surface. The spongy areas appeared and were at first thought to be regions of undissolved columbium powder. A typical shiny defect in a fractured tensile bar is shown in Figures 6 and 7. These spongy regions were often not detectable by radiographic inspection of the bars prior to tensile testing. Chemical analysis of the shiny

Figure 6: This 100X photomicrograph shows an area of shrinkage defects in an alloy bar containing 2.5 atomic percent columbium. This type of defect is also shown in a macrophoto in Figure 6. This defect commonly occurred along the central axis of columbium alloy cast bars. Such a defect has never been noted in unalloyed uranium bars cast in the same manner.

Figure 7: This macrophotograph shows the fracture surface of four standard 0.505 inch tensile bars. The bar on the right of each pair has been brightened by dipping in a nitric acid bath. The bars in the top row contain 0.9 atomic percent columbium; the pairs in the bottom row contain 1.5 atomic percent columbium. Pairs on the left are in the as-cast condition and pairs on the right have been solution treated at 850°C for $1\frac{1}{2}$ hours, followed by a water quench, and aged at 350°C for three hours and furnace cooled. The large grain size of the 0.9 atomic percent columbium alloy in the as-cast condition and the small size of the treated specimen is shown. The shiny areas of the 1.5 atomic percent alloys are shrinkage defects, also shown in Figure 5.

regions failed to show high columbium content and the regions were later proven to be caused by shrinkage. Tapered bars were cast to provide progressive solidification within the bar castings, but the defective regions also occurred in the tapered castings. The problem was overcome by casting bricks $2\frac{1}{4}$ inches by $2\frac{1}{4}$ inches by 6 inches long topped with an adequate feeder head. Each brick casting was sectioned through the vertical axis into four square bars. Any shrinkage cavities occurring in the brick were limited to the central axis region and eliminated by the saw cuts or subsequent machining of the bars to size. It was felt that tensile bars cut from brick castings more nearly represented the metal which would be achieved in a production casting, as the mass of the brick was closer to the mass of a normal casting and the cooling rates could be more closely related.

Heat Treating Procedures for Uranium-Columbium Alloys

Although the physical properties of the columbium-uranium alloys were considerably higher than unalloyed uranium in the as-cast condition, heat treatment of alloyed bars was investigated to determine what advantages might be gained. It was found that columbium alloys were highly responsive to heat treatment and that higher strengths and hardnesses could be obtained with only slight loss of ductility. The heat treatments investigated were based on procedures reported by Sawyer (4). Comparative data for as-cast and heat-treated bars are shown in Table II.

TABLE II

Kelt No.	Analysis at.% Cb	Treatment See (1)	Ultimate Str. psi	Yield Str. psi	Elong. % in 2"	R. A. %
2210	0.9	As-cast	118,000	46,500	13.3	9.9
2210	0.9	b, f	121,000	53,500	7.0	5.5
20215	1.1	As-cast	102,600	39,000	22.0	5.6
20215	1.1	b, e	106,100	50,000	16.0	8.7
2215	1.3	As-cast	120,000	42,500	16.7	7.4
2215	1.3	b, f	101,350	62,000	6.3	1.6 (2)
2215	1.3	b, g	116,000	41,000	19.8	13.4 (3)
1225	1.3	As-cast	129,000	47,100	16.4	13.8
3215	1.6	As-cast	120,030	49,500	10.2	8.2
3215	1.6	As-cast	120,970	50,500	10.2	6.7
3215	1.6	As-cast	120,490	52,500	10.2	9.5
5215	1.8	As-cast	131,520	46,000	23.5	8.4
5215	1.8	As-cast	131,800	43,500	24.2	11.6
5215	1.8	c, f	134,300	58,000	12.6	9.1
3220	1.9	As-cast	112,000	51,000	3.1	4.9 (2)
3220	1.9	b	114,460	76,000	2.3	0.8 (2)
3220	1.9	b, h	128,300	54,000	10.9	6.3 (3)
4225	2.1	As-cast	128,500	57,000	7.1	7.5
4225	2.1	a	130,000	89,000	2.4	3.9 (2)
4225	2.1	b, e	117,000	87,000	1.9	0.8 (2)
5225	2.2	As-cast	97,000	55,000	2.0	2.6 (2)
5225	2.4	As-cast	127,000	59,500	4.7	4.5 (2)
5225	2.4	b, f	95,400	87,500	Nil	Nil (2)
5225	2.4	d	95,750	77,500	0.8	0.2 (2)
7225	2.5	As-cast	115,300	58,000	3.1	2.6 (2)

- (1) Special Treatments:
- a. Cast bar cold worked by pressing
 - b. 850°C for 1½ hours and water quenched
 - c. 800°C for 1½ hours and water quenched
 - d. 700°C for 1½ hours and water quenched
 - e. 350°C for 1½ to 2 hours and furnace cooled
 - f. 350°C for 2½ to 4 hours and furnace cooled
 - g. 600°C for 1½ hours and furnace cooled
 - h. 600°C for 5½ hours and furnace cooled

(2) Tensile bar fractures showed shrinkage defects causing reduced ductility.

(3) These tensile results indicate over aging.

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Alloyed bars containing from 0.4 to 3.5 atomic percent columbium were held for varying times at 700, 800 and 850°C and quenched. The quenched bars were aged at 350°C for from 1½ hours to 4 hours followed by furnace cooling. Hardnesses and tensile tests indicated the most satisfactory treatment for columbium alloyed bars within the composition and size ranges investigated was as follows: heating at 850°C for 1½ hours followed by a water quench, and aging at 350°C for two hours with sample cooled in the furnace.

Tests were conducted on a Jominy end quench specimen of a 1.35 atomic percent columbium alloy to indicate the effective depth of a water quench subsequent to 1½ hours at 850°C. High hardness was obtained within one-half inch of the quenched end and moderately effective hardening with 1 3/4 inch of the quenched end, as shown in Figure 8. Figure 9 shows the hardness results for a Jominy end quench of an aluminum-uranium alloy bar.

Characteristics of Uranium-Columbium Alloys

Alloys containing greater than approximately 1.0 atomic percent columbium had a fine grained structure, as shown in Figures 7 and 10. One bar casting which analyzed 0.9 atomic percent columbium and all castings examined which analyzed less than 0.9 atomic percent columbium had comparatively coarse grained structures in the as-cast condition. There was indication that alloys containing 0.9 atomic percent columbium were grain refined by treatment within the gamma-uranium

SECRET

Fig. 6

Alloy: 2.13 Atomic % aluminum in uranium

Treatment: Jominy end quenched from 740°C after 1½ hours at temperature.

Hardness series indicate erratic hardenability.

Fig. 9

TREATMENT: As-cast

TREATMENT: 850°C for one and one half hours and water quench.

TREATMENT: 850°C for one and one half hours and water quench.
350°C for two and one half hours and water quench.

Figure 10: These 100X photomicrographs show structures obtained from three samples of the same casting analyzing 2.2 atomic percent columbium. The bars were subjected to the different treatments as given above. Although the structures appear similar, investigations have shown that the hardnesses and yield strengths of these alloys increase with the heat treatments given above.

Fig. 11

temperature range.

Tensile tests of sound bars revealed high physical properties. Bars analyzing approximately 1.5 atomic percent columbium and tested in the as-cast condition averaged 125,000 psi ultimate strength, 45,000 psi yield strength (0.2% offset), 10% elongation in a 2-inch gauge length, and 8% reduction in area. These values should be compared with properties reported by D. T. Doll (7) in tests conducted on unalloyed high purity uranium where values of 71,890 psi tensile strength, 28,000 psi yield strength (0.2% offset), 6.25% elongation in two inches and 3.31% reduction in area were found, and tensile properties for unalloyed uranium shown in Figure 11.

An increase in the amount of columbium, in so far as explored in this program, appeared to further increase the yield strength with a loss in ductility. Heat treatment at 850°C with aging at 350°C also raised the tensile properties of the metal.

Creep test values of columbium alloys in both the as-cast and heat treated conditions are presented in Table III. Creep data on unalloyed uranium samples are shown for purposes of comparison.

Columbium additions appeared to increase the corrosion resistance of uranium, but not to as great an extent as aluminum additions. Machined samples of columbium alloy did not form dark oxide coatings as rapidly as unalloyed uranium.

TABLE III

Room Temperature Creep Data
Uranium-Columbium Alloys and Normal Uranium 0.505 Tensile Bars

Melt No.	Cb Content At. %	Treatment	Constant Load, psi	0 hr.	12.5 hr.	25 hr.	50 hr.	100 hr.	200 hr.	300 hr.
-	0	As Cast	26,075	5.2	6.6	7.9	9.0	9.2	9.3	9.5
3215	1.6	As Cast	26,075	1.0	1.1	1.1	1.2	1.2	1.2	1.2
-	0	As Cast	31,075	5.7	8.0	8.2	8.4	8.8	9.2	9.4
5215	1.6	(1)	31,075	1.1	1.2	1.3	1.3	1.3	1.4	1.4
-	0	As Cast	36,075	12.6	16.8	17.0	17.3	17.5	17.6	17.8
15215	1.6	As Cast	36,075	2.2	2.6	2.7	2.8	2.8	2.9	2.9
20215	1.1	As Cast	36,075	2.1	-	2.7	2.8	2.9	-	-
20215	1.1	(2)	36,075	1.3	-	1.6	1.6	1.6	1.6	1.6

(1) 850°C for 1½ hours and water quenched, followed by 350°C for 4 hours and furnace cooled.

(2) 850°C for 1½ hours and water quenched, followed by 350°C for 2 hours and furnace cooled.

CONCLUSIONS

Uranium-Aluminum Alloys

1. It is difficult to achieve the desired analysis in a uranium-aluminum alloy prepared in a vacuum furnace due to vaporization losses of aluminum.
2. Uranium-aluminum alloys were not obtained with a homogeneous aluminum distribution, as is shown by widely varying analyses between the top and bottom sections of castings.
3. Aluminum alloys did not respond uniformly to heat treatment. Considerable variation in hardnesses was obtained upon solution treating in the gamma range. Solution treating in the beta temperature range generally gave more consistent hardnesses but not a sufficient increase in physical properties over the as-cast aluminum alloy to warrant heat treatment in this range.
4. Physical properties, higher than those of unalloyed uranium, were obtained with aluminum alloys but the values were erratic and difficult to reproduce due to melting problems.
5. Alloys containing greater than 2.5 atomic percent aluminum had low ductility in the as-cast and solution treated conditions.
6. The atmospheric corrosion resistance of aluminum alloys in the aluminum content ranges investigated appeared to be superior to either columbium alloys or unalloyed uranium.

Uranium-Columbium Alloys

1. In order to consistently gain complete solution of columbium in uranium at temperatures suitable for production casting techniques it is necessary to use enriched alloy biscuits prepared in a reduction bomb.
2. Carbon content of alloy castings should be held as low as possible as it has been shown that the presence of carbon promotes the flotation of columbium probably as a carbide. The flotation of the columbium causes variance in analysis throughout the castings and machining difficulties.
3. Provided the carbon content of the castings was maintained low, chemical analyses indicated the dissolved columbium to be evenly distributed within the cast alloy. Hardness values determined on columbium alloy samples indicated uniform and reproducible hardnesses.
4. To obtain tensile and creep specimens free from internal shrinkage defects, it was found necessary to cast a brick shape which subsequently sectioned through the vertical axis to yield the required bars.
5. Improved tensile and creep resistant properties were obtained from as-cast columbium alloy bars as compared to unalloyed uranium.
6. The columbium alloy bars responded to heat treatment by showing elevated yield strengths.

7. The use of an as-cast 1.5 atomic percent columbium alloy has been recommended for use.

This composition combines a relatively high yield strength and resistance to creep with a low alloy content. If higher yield strengths are desired the use of a 1.5 atomic percent columbium or a heat treatment is recommended.

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