UNCLASSIFIED

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

UNCLASSIFIED

246

WASH-701

METALLURGY AND CERAMICS (M-3679, 19th Ed.)

10

UNITED STATES ATOMIC ENERGY COMMISSION

URANIUM ALLOY NEWSLETTER

Edited by
E. Epremian

CLASSIFICATION CANCELLED

DATE MAR 13 1957 *fer*

For The Atomic Energy Commission

H. F. Canale
Chief, Declassification Branch

February 1957
[TISE Issuance Date]

Division of Research
Washington, D. C.

Price \$ 0.40

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

Technical Information Service Extension, Oak Ridge, Tenn.

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.

~~CONFIDENTIAL~~

This report has been reviewed under the Declassification Guide of 1955.

LEGAL NOTICE

This report was prepared as a part of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to any information pursuant to his employment or contract with the Commission.

This report has been reproduced directly from the best available copy.

Printed in USA. Charge 40 cents. Available from the Technical Information Service Extension, P. O. Box 1001, Oak Ridge, Tennessee. For direct to the same address inquiries covering the procurement of classified AEC reports.

~~CONFIDENTIAL~~

AEC, Oak Ridge, Tenn.

TABLE OF CONTENTS

Argonne National Laboratory	2 - 9
Armour Research Foundation	9 - 12
Atomics International	12
Battelle Memorial Institute	13 - 17
Hanford Atomic Products Operation	17
National Bureau of Standards	18 - 20
Oak Ridge National Laboratory	20 - 25
Sylvania Electric Products, Inc.	25 - 27
Westinghouse Atomic Power Division	27 - 36

The following report no new developments:

Nuclear Metals, Inc.

The following have not been heard from:

Ames Laboratory
Brookhaven National Laboratory
DuPont/Company
G. E. Co., ANP Project
Knolls Atomic Power Laboratory
Los Alamos Scientific Laboratory
Mallinckrodt Chemical Works
National Lead Company

ARGONNE NATIONAL LABORATORY - Reported by L. T. Lloyd

Uranium-Ruthenium Alloys

The U-Ru binary phase diagram is being investigated as a portion of U-Mo-Ru ternary alloy studies. The metallographic work to date has established the eutectic temperature, the first eutectoid temperature, the extent of γ solid solubility, and the existence of U_2Ru - these are shown graphically in Figure 1. The dotted lines represent the probable course of the other phase boundaries, however, they are not to be considered as correct. The crystal structure of U_2Ru (18 w/o Ru) is being investigated. It was found that an alloy containing 5 w/o Ru could be quenched from 900°C to retain body-centered-cubic γ -U with a lattice parameter of 3.46 Å, indicating that Ru as a solute is nearly as effective as Mo in contracting the lattice parameter. (Dwight)

Uranium-Silicon Alloys

Previous investigations of the corrosion behavior of the irradiated U - 3.8 w/o silicon alloy were limited to the cast material, however, if the material were used as a fuel element it is likely that it would be in a wrought form. In addition, the behavior of a defected clad structure is of interest.

The samples described here were prepared at Nuclear Metals, Inc., by heat treating at 800°C for 7 days before coextrusion with Zircaloy II tubing. The clad rod was then cut into short lengths for test. The core was exposed at the ends of each sample. Irradiation in the MTR was done by J. H. Kittel.

Unirradiated samples: A few samples were corrosion tested, in the unirradiated condition. Exposure to water was at 290°C for periods up to about 19 days. Although the samples appeared smoother than the cast material previously studied, corrosion rates remained inconsistent. Rates

UNCLASSIFIED

Figure 1

Argonne National Laboratory

UNCLASSIFIED

DECLASSIFIED

U N C L A S S I F I E D

varied erratically from sample to sample and for the same sample from period to period. The extremes were approximately 1 and 15 mg/cm²-day (mcd), based on the exposed area of the alloy. At the end of the nineteen day period some bond areas had failed while on other samples the bond area was sound. Additional heat treatment after extrusion (825°C - 7 days) resulted in increased corrosion of core and bond.

Irradiated samples: Samples were irradiated both as clad and in the stripped condition. After irradiation the stripped samples were dark black, smooth, and lustrous. As far as physical condition is concerned, they appeared to be in the best condition of any irradiated uranium alloy that has been examined prior to corrosion testing.

Results of the corrosion tests follow:

1. CE 2-1 - 0.115 total a/o burn-up: This sample was irradiated with clad removed. It was tested at 260°, 290°, and 315°C. Results are:

Temp., °C	Time, days	Average Rate of Weight Loss, mcd	Comments
260	2.5	0.44	Lustrous black except for some small white spots, probably due to incomplete removal of cladding.
290	3.4	-0.27 (wt. gain)	No change in appearance.
	2.5	7.43	Sample broken into four large pieces. "Rate" based on wt. of all pieces. Hydrogen pressure less than 25 psi.
315*	2.5	-	Sample reduced to mass of small particles. Hydrogen pressure of about 30 psi.

* Largest piece remaining after 2nd period at 290°C was tested at 315°C.

2. CE 2-2 - 0.115 total a/o burn-up: This sample was irradiated with the zircaloy clad on. After irradiation a 0.050 in. hole was drilled through the clad into the core. It was tested at 290°C and 315°C.

U N C L A S S I F I E D

There was no change in appearance after 3.1 days at 290°C. The water was clear and apparently free of oxide or metal particles.

It was then tested for 4.8 days at 315°C. At the end of this time the core was corroded about 1/32" to 1/16" below the clad at each end. The clad was swelled and cracked at an identification notch at one end of the clad. There was oxide at the drilled hole but no swelling. There were many loose particles in the water but no residual hydrogen pressure.

3. CE 1-1 - 0.098 total a/o burn-up: This sample was irradiated with the zircaloy clad removed. It was tested at 315°C for 2.9 days. At the end of the time the sample was broken into three large pieces (split in a roughly axial manner) and many small brittle pieces. The surfaces of the larger pieces were pitted. There was no residual hydrogen pressure.

4. CE 1-2 - 0.98 total a/o burn-up: This sample was irradiated with the zircaloy clad on. The core was exposed only at the ends. It was tested at 290° and 315°C.

There was no change in appearance after 3.1 days at 290°C. The water was clear and apparently free of oxide or metal particles.

After 6.2 days at 315°C there was some oxide in the water and a small chip was cracked out of the core at the identification notch in the clad. The clad was flared out at this point. There was no residual hydrogen pressure.

After an additional 7.7 days at 315°C, the core was corroded about 1/16" below the clad at both ends. Both the core and clad were cracked at the notch in the clad and this end of the sample had swelled. There was oxide in the water and a residual hydrogen pressure of about 60 psi.

5. CE 4-1 - 0.069 total a/o burn-up: This sample was irradiated with the zircaloy clad removed. It was tested at 315°C for 10.6 days.

U N C L A S S I F I E D

At the end of this period it was reduced to a slurry. There was a residual hydrogen pressure of 40 psi.

6. CE 3-2 - 0.085 total a/o burn-up: This sample was irradiated with the zircaloy clad on. After irradiation, a 0.050 in. hole was drilled through the clad into the core.

It was tested at 315°C for 6.6 days. At the end of this time the core was corroded about 1/16 in. below the clad at each end. Both core and clad were cracked at the notched end of the clad and the sample was swelled at this end. There was no swelling at the drilled hole. There was oxide in the water and a residual pressure of about 25 psi.

Conclusions: Bare U - 3.8 w/o Si alloys, after irradiation, fail after a relatively short exposure to high temperature water. The failure may not be solely the result of corrosion. The effect of partial cladding is difficult to evaluate because all clad samples were mechanically damaged in the process of identification. In order to determine the effect of cladding it would be desirable to have completely clad samples with small defects.

It is evident, based on the results of the unirradiated samples, that the lack of uniformity of these alloys is still a major problem.
(Draley and Greenberg)

Uranium-Titanium Alloys

Samples of a γ quenched U - 3 w/o Ti alloy, aged at 400°C for periods of 1 to 4 hours, were completely disintegrated after corrosion for one week in water at 290°C. (Draley and Greenberg)

Uranium-Zirconium Alloys

The U - 2 w/o Zr alloy is of interest because of its dimensional stability under nuclear radiation. It does not, however, have aqueous corrosion resistance, even at 100°C.

U N C L A S S I F I E D

Samples of the alloy were coextruded with Zircaloy II tubing and completely clad samples prepared by welding in plugs of Zircaloy II at the bored out ends. In an effort to produce a corrosion-resistant barrier layer, the samples were diffusion heat treated at $1050^{\circ} \pm 5^{\circ}\text{C}$ for two hours and air cooled.

To test the efficiency of the diffusion produced barrier layer, a 0.005 in. hole was drilled through the clad and just barely to the core (on the cylindrical surface). On some samples the hole was drilled before the diffusion heat treatment; on others, after the treatment.

The idea for the diffusion heat treatment originated at Nuclear Metals, Inc. (where the samples were prepared) and the samples were tested as a service to this organization. Results follow:

Unirradiated Samples:

1. Sample A-200 was stripped of its cladding after the diffusion heat treatment. It was corrosion tested at 290°C for 1.7 days. At the end of this time it was completely reduced to an oxide slurry.

2. Samples A-138 (defected after heat treatment) and A-175 (defected before heat treatment) were tested for 8.8 days at 290°C and then for an additional 13 days at 350°C . At the end of this test period there was no apparent change in the appearance of the samples, except that Sample A-138 developed a white area around the drilled hole. The welded plugs were white upon receipt of the sample, presumably from prior corrosion testing.

The samples were then sectioned through the drilled hole and through the clad-core interface near the welded plugs. There was no evidence of core corrosion.

3. Samples A-137 (defected after heat treatment) and A-145 (defected before heat treatment) were tested for 21.8 days at 290°C and

then for 31.7 days at 350°C. At the end of this time there was no change in dimensions or appearance except for the development of a small white area around the hole in A-145.

Irradiated samples: These samples were irradiated prior to corrosion testing (by J. H. Kittel in the MTR). Burn-ups of approximately 0.1 total atom percent were obtained.

Sample A-174 (CH-1C; defected before heat treatment) was tested for 2.5 days at 290°C and then for 9.0 days at 315°C. Sample A-141 (CH-2F; defected after heat treatment) was tested for 2.6 days at 290°C and then for 9.2 days between 315° and 338°C. At the end of the given test periods there were no apparent changes in the samples.

Conclusion: The diffusion heat treatment definitely produced a protective barrier under the conditions of test. This protection is apparently unaffected by relatively small amounts of nuclear radiation. It would be desirable to test material subjected to higher burn-up. (Draley and Greenberg)

Uranium-Zirconium-Niobium Alloys

The study of the mechanism of corrosion resistance of the U - 5 w/o Zr - 1.5 w/o Nb alloy is continuing. There is some evidence (A. E. Dwight) that rapidly quenched specimens of this alloy are single phase structures. According to our present concepts such a material would not be expected to be corrosion resistant, unless the martensitic structure has a high rate of permeation by hydrogen.

Tests on rapidly quenched (and apparently single phase) small samples have shown corrosion behavior identical to the larger specimens previously tested. This result makes it necessary to believe either (a) there is a submicroscopic phase present, or hydrogen diffuses readily into the martensite; or (b) our theory, requiring the absorption

of hydrogen into the surface of these "good" alloys during corrosion, does not apply. (Draley and Greenberg)

Uranium-Zirconium-Tantalum Alloys

An ingot of U - 5 w/o Zr - 2 w/o Ta was not corrosion resistant at 290°C in the cast condition after quenching from 1070°C. When this material was rolled and quenched it was corrosion resistant for a relatively short time. Aging the rolled and quenched alloy at 400°C for periods of 2 and 4 hours destroyed its corrosion resistance at 290°C (complete disintegration and a rate of 119 mcd, respectively after 3.7 days). The results for the quenched and quenched and aged 400°C - 1 hour are summarized below.

Aqueous Corrosion of U - 5 w/o Zr - 2 w/o Ta at 290°C

Test Interval, days	Average Rate of Wt. Loss During Interval, mcd	
	Quenched from 1070°C	Quenched & Aged at 400°C - 1 hr.
3.75	16.1	14.1
6.0	31.6	230

The test was stopped after 9.75 days. Attack was non-uniform and apparently two types of oxide (black and reddish brown) were formed. It is apparent that the problem of homogeneity in uranium-tantalum alloys has not been solved. (Draley and Greenberg)

ARMOUR RESEARCH FOUNDATION - Reported by R. J. Van Thyne

Transformation Kinetics of Binary and Ternary Uranium-Niobium Alloys

Isothermal transformation kinetics of the gamma phase of U-Nb and U-Nb-X alloys are under investigation. The experimental techniques employed have been described previously. Most of the compositions (w/o) are shown in Figure 1. In addition, alloys containing 7 w/o Nb, 7 w/o Nb-1 w/o Zr, 10 w/o Nb-0.13 w/o Cr, 10 w/o Nb-0.78 w/o Cr, 8 w/o Nb-0.12 w/o Cr, 8 w/o Nb-0.84 w/o Ni are being studied. With the exception of the U-Nb-Zr alloys, the ternary additions were added to the 8 or 10 w/o Nb

base composition on the basis of atomic percentages and represent 0.5 to 8 a/o of the various elements.

The annealing time required to produce initial transformation in all alloys at 500°C was under 4 hours. Hardness, resistivity and metallographic results were in close agreement. At 450°C decomposition at the grain boundaries of the various alloys was detected between 0.1 and 100 hours. As described in the last Newsletter, a fine precipitate was generally observed in the grains prior to the appearance of the grain boundary transformation. A resistivity change was observed before decomposition was noted by any other technique.

Transformation occurred at the grain boundaries in the U-8 w/o Nb-0.68 w/o Si alloy upon annealing at 360°C for 4 hours. All other alloys required at least 100 hours for such a structural change and no decomposition at the grain boundaries was detected for some compositions after 1000 hours at 360°C. Other structural and physical property changes occurred in shorter times than those required for the grain boundary decomposition. Apparently a pre-precipitation phenomenon, a hardness increase always resulted at times less than those required for changes in other properties at 360°C.

Results of hardness measurements (Figure 1) show the effects of the various ternary additions to the U-Nb base material. The annealing time required for an initial hardness change was increased by additions of titanium, nickel and silicon. Chromium and ruthenium appear to have a moderate stabilizing effect and vanadium additions retard hardness changes considerably. Hardness increases occurred most rapidly at the 550°C annealing temperature for all compositions except those containing zirconium, where the nose of the TTT curves was noted at 450°C. U-Nb-Si alloys transformed very rapidly due to the formation of an intermetallic compound rich

UNCLASSIFIED

ARMOUR RESEARCH FOUNDATION
Figure 1

FIG. 1 - TTT DIAGRAMS ILLUSTRATING INITIAL HARDNESS CHANGE FOR U-Nb AND U-Nb-X ALLOYS

UNCLASSIFIED

U N C L A S S I F I E D

in niobium, which lowered the niobium content of the matrix.

X-ray diffraction studies have shown that for most alloys, transformation proceeds by a continuous precipitation of alpha from the metastable gamma phase. Spectrometer traces show that as increased amounts of alpha are formed, the gamma peak shifts to higher 2θ values due to niobium enrichment. A series of patterns for the U-7 w/o Nb-2 w/o Zr alloy annealed at 550°C showed that at one stage of transformation, the gamma peak became low and poorly defined, probably the result of a finely dispersed gamma structure having a range of compositions. Subsequent annealing, resulting in agglomeration and diffusion, caused the gamma reflection to become more sharply defined. The location of the gamma peak in the patterns approached the equilibrium values for γ_2 .

ATOMICS INTERNATIONAL - Reported by B. R. Hayward

The irradiation of samples of SRE fuel materials have been completed in the MTR. The peak temperature measured at the center of the $3/8'' \times 1\frac{1}{2}''$ specimens was 1080°F . The NaK bonded slightly enriched (10%) specimens in duplicate were alpha rolled beta treated uranium. U-Mo (1.2 w/o Mo) powder compacted, and U-Zr (2.0 w/o Zr) cast. Calculated burnup of the specimens was 0.5% of total atoms. Shipment of samples is in progress and examination in the SRE hot cell is planned for February. A second series of tests has completed 0.2% burnup (out of 0.5% planned) at MTR with a peak central measured temperature of 1225°F .

U N C L A S S I F I E D

0370000000

BATTELLE MEMORIAL INSTITUTE - Reported by F. A. Rough

Constitution

High-Temperature X-Ray Diffraction Examination of the Uranium-Carbon System - Satisfactory samples for use in the high-temperature x-ray camera have been prepared using the combustible-plastic-capillary technique. Both UC_2 and UC samples have been prepared to date and the U_2C_3 compositions are presently being prepared.

The UC_2 was examined at high temperatures in the diffraction camera and a transformation was observed at $1800^\circ C$ to a face-centered-cubic phase of a nominal lattice parameter of 5.47 Å. The transformation is reversible and was confirmed by direct test on a second sample. A second transformation of UC_2 is currently being investigated and is suspected to form a phase intermediate between room-temperature UC_2 and the high-temperature phase.

Transformation Studies

Transformation Kinetics of Zirconium Alloys Containing 20 to 70 w/o Uranium - The study of the kinetics of the beta-to-epsilon decomposition in Zr-U alloys is continuing.

Dynamic modulus measurements have been completed on a series of specimens of the 50 w/o uranium alloy. These specimens were quenched after 1 hour at $800^\circ C$ and transformed for 6 minutes, 1 hour, and 16 hours, respectively, at temperatures ranging from 100 to $500^\circ C$. The data are found to fit an equation of the form $F = 1/2 [1 - \text{erf} \frac{A}{T} + B]$, where, if it is assumed that the changes in dynamic modulus are proportional to transformation, F = fraction of beta transformed, and T = temperature. The constants, A and B , are being evaluated at present. An activation energy of 27,600 cal/mole is estimated for the transformation.

~~CONFIDENTIAL~~

Some tentative conclusions may be drawn on the basis of the above data and the work of previous months. First, the reaction is diffusion controlled, and second, while a hardening reaction is associated with the transformation, maximum hardness does not represent the completion of transformation.

Alloy Development and Radiation Effects

Uranium Alloys for High-Temperature Application - This program is being continued with the effort presently concentrated upon the determination of the fabricability of the uranium-20 w/o niobium alloy, and upon the preparation of enriched-uranium irradiation specimens of uranium-15 w/o zirconium and uranium-10 and 20 w/o niobium. More details of this program will be reported as further progress is made.

Preparation and Properties of Low-Melting Alloys - This program is concerned with evaluation of an uranium-5 w/o chromium-1 w/o molybdenum alloy for use as a power-reactor fuel.

Machined pins of as-cast alloy are being encapsulated for irradiation at temperatures of 600°F and lower. The specimens will be irradiated to approximately 1 a/o burnup and examined for neutron-damage effects.

Radiation Stability of Zirconium-Uranium Alloys - Work is being continued on the radiation-effects program for the zirconium-22 and -50 w/o uranium alloys.

Three non-temperature-controlled capsules, BMI 8-1, BMI 8-2, and BMI 8-3 have been irradiated at the MTR in an integrated flux of about 6.2×10^{20} nvt (nominal flux of 0.8×10^{14} nv for 89.9 days). Each capsule contained three specimens of fully enriched zirconium-22 w/o uranium alloy 1 inch long and 0.100 inch in diameter, and each specimen was in a small individual capsule containing NaK. The estimated core

~~CONFIDENTIAL~~

CONFIDENTIAL

temperature of each specimen was 400°C. The calculated burnup for all nine specimens is 1.95 a/o total. Capsules BMI 8-2 and BMI 8-3 have been returned to the hot-cell facility and the post-irradiation investigation of these specimens was begun in December. The objective is to evaluate the effects of varying microstructure on radiation performance. Capsule BMI 8-1 has not been received from the MTR.

A temperature-controlled capsule which contained three specimens of fully enriched zirconium-22 w/o uranium alloy operated successfully for 19.3 days in a nominal flux of 0.9×10^{14} nv. At the end of this time, an accident occurred at the MTR and the power leads to the capsule were severed, causing irradiation for about nine MTR cycles to obtain a burnup in the specimens of about 2 a/o total; however, it is now planned to ship the capsule to BMI and examine the specimens. These three specimens were irradiated to an estimated burnup of about 0.43 a/o total at a core temperature of 675°C.

Corrosion

Development of Corrosion-Resistant 70 w/o Uranium Alloys - An investigation aimed at the development of a corrosion-resistant 70 w/o uranium alloy is being made. The zirconium-70 w/o uranium alloy is serving as the base alloy for this study.

Two approaches are being studied as possible means of improving the corrosion resistance of the base alloy, heat treatment and ternary alloying. The primary purpose of ternary additions is to tie up the alpha-uranium phase which precipitates from the zirconium-70 w/o uranium alloy in the form of a compound or intermediate phase. Upon the basis of previous experimental work, molybdenum, silicon, and titanium have been selected for this purpose.

~~CONFIDENTIAL~~

TABLE 1. EFFECT OF TEMPERATURE AND VARIOUS ZIRCONIUM MELTING STOCKS ON THE CORROSION BEHAVIOR OF ZIRCONIUM-URANIUM ALLOYS IN HIGH-TEMPERATURE WATER

Nominal Uranium Content (Balance Zirconium), w/o	Zirconium Melting Stock	Weight Change Rate (a), mg/(cm ²)(hr) After 3024 Hr of Exposure at Temperature Indicated		
		500 F	600 F	680 F
45	Crystal bar	(b)	-0.043	-0.11
	Sponge	-0.003	-0.051	-0.14
	Sponge + 0.3 w/o O	-0.004	-0.057	-0.15
50	Crystal bar	-0.001	-0.062	-0.15
	Sponge	-0.003	-0.069	-0.17
	Sponge + 0.3 w/o O	-0.005	-0.084	-0.21
55	Crystal bar	-0.003	-0.069	-0.15
	Sponge	-0.003	-0.080	-0.18
	Sponge + 0.3 w/o O	-0.006	-0.096	-0.25
60	Crystal bar	-0.003	-0.066	-0.19(a)
	Sponge	-0.005	-0.093	-0.26(c)
	Sponge + 0.3 w/o O	-0.015	-0.19	-0.64(c)
70	Crystal bar	-0.36(c)	(d)	(d)
	Sponge	-0.71(c)	(d)	(d)
	Sponge + 0.3 w/o O	-1.23(c)	(d)	(d)

(a) Calculated when rate change became linear with time.

(b) Gained weight; little weight change over last 1512 hr of exposure.

(c) Samples cracked or completely oxidized prior to 2520 hr of exposure.

(d) Completely oxidized within a 168-hr exposure.

BATTELLE MEMORIAL INSTITUTE

Table 1

~~CONFIDENTIAL~~

0370291030

Heat treatment of the zirconium-70 w/o uranium alloy has been found effective in improving corrosion resistance, isothermal transformation of the beta-zirconium phase at 500°C for about 100 seconds markedly increasing corrosion life in 680°F water. Additional specimens are being heat treated for verification of these results and for corrosion tests in other heat-treated conditions. Similarly, ternary alloys have been prepared for corrosion test in a variety of heat-treated conditions. Included are quench, quench-and-temper, isothermal-transformation, and furnace-cooling treatments.

HANFORD ATOMIC PRODUCTS OPERATION - Reported by S. H. Bush (Unclassified)

Thorium-2 w/o U-235 Irradiation

Two samples of Th-2 w/o U-235 alloy have been visually examined and bend-tested after irradiation to calculated exposure of 6×10^{20} nvt (5,000 MWD/T or 0.5 a/o burnup) and 15×10^{20} nvt (10,000 MWD/T or 1.0 a/o burnup). Visual examination reveals good dimensional stability of the alloy under conditions of the irradiation (i.e., heavy-walled restraining jacket). Unirradiated control pieces bent on one inch centers to the limit of the test apparatus at about 3500 pounds load without fracture. Each of the irradiated samples fractured at about 5000 pounds load but with only 0.004-inch deflection. The strength of the material has increased, but the ductility has been markedly reduced. X-Ray study of this material for lattice distortion resulting from the burnup is being planned. Additional samples of the alloy are being irradiated to planned exposures up to 3.0 a/o burnup. Some of these samples are being irradiated in NaK containing capsules, hence are physically unrestrained. The dimensional stability of the unrestrained pieces at high burnups will be of great interest. (J. W. Goffard) (Unclassified)

NATIONAL BUREAU OF STANDARDS - Reported by J. J. Park

Uranium-Iridium System

The addition of iridium to uranium lowers the gamma-beta and the beta-alpha transformation temperatures of uranium, though the temperature of transformation is somewhat dependent upon the rate of cooling. When cooled at $3^{\circ}\text{C}/\text{min}$, the gamma-beta transformation occurs at 750°C and the beta-alpha transformation occurs at 534°C . When cooled at $0.8^{\circ}\text{C}/\text{min}$, the gamma-beta transformation is at 750°C and the beta-alpha transformation is at 560°C .

Uranium-Rhodium System

Results of thermal analysis runs on alloys containing up to nominal 50 w/o of rhodium provide data for some tentative conclusions concerning the uranium-rich portion of the phase diagram. The addition of rhodium to uranium lowers the melting point of uranium to about 850°C with the formation of a eutectic at about 12 w/o of rhodium. The uranium transformation temperatures are lowered by the addition of rhodium, the gamma-beta transformation occurring at about 700°C and the beta-alpha transformation occurring at about 625°C . The persistence of the uranium transformations with increasing amounts of rhodium leads to the postulation of an intermetallic compound at 50 a/o of rhodium. This compound is apparently formed at about 1350°C .

Uranium-Osmium System

The absence of the beta-alpha transformation arrest during the thermal analysis cooling run of alloys with less than 5 w/o of osmium indicated that beta-uranium might be retained at room temperature by quenching techniques. However, x-ray diffraction patterns of a 2 w/o osmium alloy quenched from various temperatures showed the presence of alpha-uranium. Other quenching treatments will be undertaken with alloys of low osmium content.

The beta-alpha transformation is not detected when low percentage osmium alloys are investigated by thermal analysis. Heating the same alloy for a period of time below the gamma-beta transformation temperature will bring out a strong beta-alpha transformation at 475°C when cooled at $3^{\circ}\text{C}/\text{min}$. The variation of transformation temperature with cooling rate will also be investigated.

Uranium-Ruthenium System

Alloys of high ruthenium content have melting points in excess of 2100°C as indicated by thermal analysis and melting point readings. An arrest was noted at about 1830°C on the heating curve of a nominal 90 w/o ruthenium; and this arrest is believed to be a reaction horizontal rather than a melting point. Metallographic observation of this sample showed a breakdown of the arc-melted structure. Back-reflection x-ray techniques were employed on the 90 w/o ruthenium alloy, and these showed a change in lattice parameter of ruthenium indicative of solid solubility of uranium in ruthenium. The extent of this solubility has not been determined.

Uranium-Palladium System

Thermal analysis results on alloys containing from 50 to 90 w/o of palladium give an indication of the liquidus curve for this system. The liquidus rises smoothly from about 1000°C at 20 w/o palladium to about 1072°C at 50 w/o palladium, rises sharply to about 1332°C at 53.6 w/o palladium, and reaches an apparent maximum of about 1640°C at 57.4 w/o (75 a/o) palladium. From this maximum the liquidus temperature decreases smoothly to a minimum of about 1350°C in the vicinity of 70 w/o palladium, rising smoothly from this minimum toward the melting point of palladium at 1552°C . Reaction horizontals are apparently indicated by the shape of the liquidus curve, though the expected horizontals have

Metallographic examination of homogenized alloys containing from 47.6 w/o to 95 w/o palladium indicates a two phase region at 47.6 and 50 w/o, a single phase region at 50 and 57.4 w/o, and a two phase region at 59.8 and 64.2 w/o. Alloys containing greater than 80 w/o of palladium were single phase, indicating a broad solid solution field at the palladium end of the diagram.

Results of Recent Investigations of Uranium-Aluminum Alloys Containing 25 to 50 w/o Uranium

An investigation is currently being conducted to obtain pertinent physical and mechanical property parameters for U-Al alloys containing 25 to 50 w/o uranium. The objective of this program is to ultimately produce an alloy, enriched 20% in the U-235 isotope, which will be suitable for manufacturing aluminum plate-type fuel elements for foreign research reactors.

Preliminary physical property data which include coefficient of linear expansion, thermal conductivity, hardness, and tensile properties both at room temperature and elevated temperature are summarized in ORNL, Table I.

The work hardening on cold rolling of nominal 25, 35, and 45 w/o U-Al alloys was also evaluated for materials having three different fabrication treatments. These treatments were:

- A. Specimens 25-A, 35-A, and 45-A were reduced 50% at 600°C by rolling and vacuum annealed six hours at 600°C prior to cold rolling.
- B. Specimens 25-B, 35-B, and 45-B were reduced 80% at 600°C by rolling and vacuum annealed prior to cold rolling.

U N C L A S S I F I E D

TABLE I

Physical Properties of Uranium-Aluminum Alloys Containing 25 to 45 wt % Uranium

I. Coefficient of Linear Expansion:

Material: As-cast 34.5 wt % U-Al Alloy

$$\delta = 21.7 \times 10^{-6}/^{\circ}\text{C}$$

II. Thermal Conductivity:

Material: As-cast 34 wt % U-Al Alloy

$$K_{200^{\circ}\text{C}} = 0.191 \text{ cal/cm}^2/\text{cm}/^{\circ}\text{C}/\text{sec.}$$

$$K_{300^{\circ}\text{C}} = 0.213 \text{ cal/cm}^2/\text{cm}/^{\circ}\text{C}/\text{sec.}$$

$$K_{400^{\circ}\text{C}} = 0.234 \text{ cal/cm}^2/\text{cm}/^{\circ}\text{C}/\text{sec.}$$

III. Hardness-Rockwell "H":

Material: Air induction melted castings, reduced 50% at 600°C
and vacuum annealed six hours at 600°C

<u>% Uranium in Alloy</u>	<u>Hardness R_H</u>
25.0	68
35.3	77
43.4	92

IV. Tensile Properties at Room Temperature:

Material: Air induction melted slab castings reduced 83% at
600°C by rolling and vacuum annealed six hours at
600°C

<u>Uranium Content</u> <u>wt % U</u>	<u>Yield Strength</u> <u>psi</u>	<u>Tensile Strength</u> <u>psi</u>	<u>Elongation</u> <u>%</u>
24.7	7,820	16,800	14.0
34.1	10,600	18,900	3.7
44.2	14,600	21,600	0.5

Results represent average of four tests on standard sheet specimens
0.088-in. thick.

OAK RIDGE NATIONAL LABORATORY
Table I

U N C L A S S I F I E D

U N C L A S S I F I E D

U N C L A S S I F I E D

TABLE I (Con't)

V. Tensile Properties at 540°C

Material: Air induction melted cylindrical castings reduced
94% at 535°C by extrusion

<u>Uranium Content</u> <u>wt % U</u>	<u>Yield Strength</u> <u>psi</u>	<u>Tensile Strength</u> <u>psi</u>	<u>Elongation</u> <u>%</u>
39.2	1,790	2,180	22.0
42.7	1,500	1,660	12.5
43.6	1,560	2,000	11.5

Results represent single test on 0.357-in. diameter specimen.

OAK RIDGE NATIONAL LABORATORY
Table I (Cont'd.)

U N C L A S S I F I E D

03712001030

23

UNCLASSIFIED
ORNL-LR-DWG 15303

Work Hardening of Uranium-Aluminum Alloys.

UNCLASSIFIED

TABLE II

Homogeneity Results in 5-1/4 x 9 x 1-in. Air Induction Melted Slab Castings of
Nominal 48 wt % U-Al Alloy

Slab No.	Average Analysis wt % U	Maximum Deviation - wt % U	
		Plus	Minus
D-584	46.90	0.54	0.46
D-585	47.20	0.44	0.61
D-595	46.13	0.85	0.49
D-596	47.08	0.55	0.35
D-601	45.71	0.69*	1.61
D-605	47.31	2.29	2.50
D-606	45.54	0.15	0.43
D-607	45.99	0.45	0.57

* Average of 6-7 analyses

OAK RIDGE NATIONAL LABORATORY
Table II

UNCLASSIFIED

0370201030

C. Specimen 45-C was reduced 94% at 535°C by extrusion and vacuum annealed prior to cold rolling.

Results of these experiments are presented in ORNL, Figure 1. These data illustrate the expected decrease in work hardening with increasing uranium content. It also appears that previous fabrication history has little effect on the work hardening of a given alloy.

Since uranium is a question of paramount importance in uranium alloys for fuel elements, the segregation in a series of 48 w/o U-Al alloys was evaluated. The uranium exhibited a marked tendency to concentrate in the upper portions of the cast slabs. By cropping and discarding about 15% of the material at the top of the casting, a reasonable homogeneous material was obtained. Data on the homogeneity of recent castings are summarized in ORNL, Table II.

SYLVANIA ELECTRIC PRODUCTS, INC. - Reported by F. E. Bowman

During the course of investigating the effects of specimen diameter upon alpha-beta cycling stability, a size dependence is indicated in the case of the 0.5 w/o and 1.0 w/o chromium alloys. It is felt that this may be related, in part at least, to the effects of chromium on the beta transformation rate. The more rapid cooling of the smaller specimens may permit the passage through the temperature range in the vicinity of the nose of the TTT curve without transformation. Consequently, the cycle employed (4 minutes cooling and 11 minutes heating) between 200°C and 700°C, does not permit a phase change and the material remains beta throughout the test, thus eliminating the distorting effects of the density change involved in the transformation as well as the purely thermal stresses which would result. That such an effect is related to the transformation rate reduction is also indicated by the essentially complete stability of the 6.0 w/o molybdenum alloys. In this case, the gamma phase remains stable

throughout the cycle.

In view of these indications of the relationship between phase transformation rate and elevated temperature cycling stability, an additional criteria is suggested for a thoroughly dimensionally stable alloy. Since TTT curves have not been determined for the molybdenum alloys in the 1.5 w/o to 2.5 w/o range, and since this composition range appears particularly attractive from the dimensional stability standpoint, the necessary data are being obtained.

In addition to the stability in thermal cycling imparted by phase stabilization, another advantage can be visualized. The swelling which has been encountered in irradiation tests which have been conducted at temperatures near the transformation range in uranium and some of its alloys, has been attributed to released fission gases which accumulate at grain boundaries, inclusion interfaces, and other structural discontinuities. Post irradiation experiments at KAPL have indicated that grain boundary movement is largely responsible for freeing the fission gases from their interatomic locations. A phase transformation certainly results in the maximum shifting of grain boundaries and would thus contribute most heavily to fission gas movement. Consequently, the elimination of phase transformations during the life of a fuel materials in a reactor should minimize this undesirable swelling which could very well be the limiting factor in the fuel life.

Alpha-beta cycling of the ternary alloys which are composed of a strengthening solid solution element and a compound-forming element in uranium is continuing. Sufficient results are not, as yet, available for a statistical evaluation of the effectiveness of this approach to stability. However, certain compositions appear to be promising. Strength data and transformation rate information are also being obtained on these materials.

A program in cooperation with Argonne has been undertaken to investigate the powder metallurgy approach to the fabrication of Al-U alloy fuels containing a homogeneous distribution of a burnable poison. There will be more to report on this subject in the next Newsletter. (Unclassified)

WESTINGHOUSE ATOMIC POWER DIVISION - Reported by J. Hino (Confidential)

Uranium-Niobium Alloy

Progressive Solidification Experiments of U-10 w/o Nb Alloys -

Experiments on the U-10 w/o Nb alloy have been initiated in an attempt to discover which and to what degree the major impurities (O, N, C) are carried along when niobium is moved to one end of the sample by progressively solidifying the molten alloy.

Work was carried out in a vertical vacuum induction furnace at 1500°C in a vacuum of $<0.5\mu$. The first sample (U-10 Nb, 3/8" diam. x 6" long) was melted in a beryllia washed graphite crucible and progressively solidified from bottom to top at the rate of 4.5 inches per hour. The ingot was removed from the crucible, top and bottom removed for metallographic examination and the remainder cut into one-inch sections. Each section was analyzed for Nb, C, O, and N. Results from this first run can be seen in Figure 1. Each point is the average value over the one-inch section.

As Nb moved to the top of the ingot, the C and O moved along with the Nb. However, there was no segregation of the N.

Two more samples have been run to determine the effect of gravity on the movement of Nb and impurities and the contribution of the liquid-solid interface movement.

One of these ingots has been run under the identical conditions described above except that the ingot was solidified from top to bottom. The other control sample was held at 1500°C for approximately 1½ hours and then allowed to solidify in the crucible. These samples are now being analyzed.
(P. W. Patterson)

Effect of Carbon on Corrosion of U-10 w/o Nb - A study has been completed concerning the influence that carbon impurities and thermal treating have on the corrosion rate of U-10 w/o Nb in pressurized water at 680°F. Four ingots of U-10 Nb containing approximately 268 ppm, 395 ppm, 832 ppm, and 962 ppm were used. The specimens from each ingot were heated to 700°C, 800°C, 900°C, 950°C, 1000°C, and 1050°C for one hour followed by a quench to room temperature. All specimens were quenched at a rate fast enough to retain the metastable gamma phase. Other samples in the "as-rolled" condition were also corrosion tested.

An exposure period of 18 days was necessary to cause complete deterioration. Examination of the specimens was made every three days and samples that were chipped or cracked were removed from the test cells.

Some general conclusions may be drawn from these studies. The weight loss is much greater per unit time the higher the carbon content. The loss was approximately three times greater between the highest and lowest carbon specimens for any one period of time. A greater percentage of high carbon pieces were also removed from test during the early stages of testing due to chipping and cracking. All samples in the as-received condition and those heat treated at 1050°C generally lost more weight and deteriorated more rapidly than those at the lower temperatures. Samples with low carbon and given a 700°C heat treatment had the lowest weight loss and were the last group to fail.

Hardness measurements were also made on specimens from each of the four ingots. Identical heat treatments as those given the corrosion specimens were given individual pieces.

A progressive increase in hardness with decrease in carbon content was noted. The influence of quenching temperature on the hardness could not be clearly established due, perhaps to inhomogeneity of carbon and niobium in the ingots. (H. Maier)

Effect of Stress and Cold Work on the Transformation of a U-10 w/o Nb

Alloy - Cold work has a very pronounced effect upon transformation on U-10 w/o Nb alloy from the metastable γ to the $\alpha + \gamma_2$ state. On samples which were cold rolled 10, 14½, 16½, 26, and 47% in one mil steps, both x-ray diffraction and metallography showed that transformation was present in samples having as low as 16½% cold work. The transformation, which is normally found along grain boundaries, is found chiefly on the slip planes in cold worked samples.

Stress also tends to hasten transformation in samples of U-10 w/o Nb alloy in the gamma state. Preliminary work on tension samples showed that stressing to 50,000 * psi at 325°C for 48 hours in a creep-machine produced an elongation of 2%, whereas stressing to 50,000 psi at 250°C for 120 hours produced an elongation of 1.3%. The latter sample shows about 3% transformation which is chiefly in grain boundaries in contrast to the cold worked samples. According to the TTT curve for this alloy, no transformation should be present. X-ray results indicate some preferred orientation of either alpha or gamma phase. However, further work is planned to verify the effect.

Preliminary work on a compression sample showed that stressing at 72,000 psi for 18 hours at 300°C in a creep machine produces a reduction of 2% in the length of the sample.

Samples which were stressed in a cantilever beam type jig showed a very strange effect. The stressed samples relaxed and actually continue to deflect away from the loading screw, to over twice the amounts of the original deflections applied to the samples. The amount of transformation is small and exists primarily on the high stress end. Permanent sets were noted on samples which were stressed to a maximum fiber stress of 108,000 psi at a temperature as low as 75°C for 120 hours and on samples stressed to as low as 8500 psi maximum fiber stress and transformed at 360°C for 24 hours. However, the permanent set was not greater than original deflections on samples transformed at 150°C for 102 hours and at 75°C for 120 hours. In all probability, these stressed samples transformed at temperatures below 300°C would eventually deflect from the screw which originated the stress if transformed for longer times. It was found that samples

* Yield stress @ 360°C ~ /20,000 psi

transformed under stress at temperatures of 550°C and above gave a permanent set equal to the original deflection, whereas those transformed between 300 and 400°C gave permanent sets greater than the original deflections. It also appears that the stressed samples attain larger permanent sets at 300 and 360°C than at 400°C. It is believed that this deflection beyond the screw which originated the stress is caused by transformation. Probably the precipitation and growth of alpha in a preferred orientation relieves the stress and further oriented growth continues to deflect the specimen. To further test this hypothesis, a sample which was stressed and transformed at 400°C for 24 hours was regammated at 900°C, and it was found to straighten to its original shape.

Electron microscopy of transformation products of U-10 w/o Nb at the stressed end of a cantilever specimen transformed at 550°C for 1½ hours showed serrations on individual lamellae. This is believed to be a result of the application of stress since unstressed transformation products do not exhibit such serrations. An attempt to confirm this effect will be made. The time dependence of deflection in the cantilever beam type specimen is being studied. The first results were obtained at 300°C and are presented in the graph (Figure 3). Relaxation is initially rapid and tends to level off somewhat after the specimen has deflected beyond the loading screw. (H. Young)

Dynamic Modulus - An unusual positive temperature coefficient of the elastic modulus between room temperature and 170°C has been observed for the gamma U-10 w/o Nb alloy. The room temperature modulus is apparently affected by the cooling rate from 170°C as indicated below.

~~CONFIDENTIAL~~

Cooling from 170°C (Orig. Gamma-Quenched)	Approx. Rate (°C/hr)	Modulus at 25°C (10 ⁶ psi)
Slow cool	3	10.35 to 10.33
Furnace cool, vacuum	30	10.25 to 10.22
Furnace cool, argon	60	10.16
Water quench	200	10.12 to 10.11

It is interesting to note that the modulus of gamma-quenched U-10 Nb at room temperature changes during aging at room temperature. It increases from 9.2 (10)⁶ psi at 6 min. after quenching to 9.8 (10)⁶ psi at 80 hours after quenching, and further to 10.17 (10)⁶ psi after 1084 hours. From two hours to 1084 hours, a plot of modulus versus log time results in a straight line.

The effect of isothermal treatment on the dynamic modulus at temperature is also being studied at 170°C, 305°C, and 360°C. A sigmoidal curve with a long linearly rising portion is found in each case. This increase in modulus is believed to be due to the gamma-alpha transformation. For example, at 360°C, the curve indicates the beginning of transformation after 30 min. at temperature.

A similar curve was constructed from room temperature modulus data taken on one specimen which was annealed various times at 500°C. Here too, beginning of transformation is indicated after about 30 minutes at temperature.

An attempt was made to study the kinetics of the change from a positive temperature coefficient to a negative (normal) coefficient of the elastic modulus. A gamma-quenched specimen was repeatedly heated to 300°C and cooled to room temperature within the dynamic modulus apparatus after holding the specimen at 300°C for 2.5 hours. The plot of modulus versus temperature throughout the history of this experiment

~~CONFIDENTIAL~~

0370201030

~~CONFIDENTIAL~~

33

is not amendable to a simple analysis or interpretation. Apparently, two opposing temperature dependent mechanisms are active. The first is the reversible and unexplained mechanism causing a positive temperature coefficient, while the second is caused by the irreversible mechanism of alpha precipitation causing a negative temperature coefficient. A temperature of about 200°C appears to separate the range below which the reversible mechanism is most pronounced, and above which the irreversible mechanism is predominant. (E.S. Wright, F.Forscher)

~~CONFIDENTIAL~~

DECLASSIFIED

UNCLASSIFIED

WESTINGHOUSE ELECTRIC CORPORATION

Figure 1

CURVE NO. 1

SIGNATURE

P. R. Miller

DATE 10/16/55 CURVE NO. 1

UNCLASSIFIED

Figure 2

Westinghouse Atomic Power Division

Figure 2

UNCLASSIFIED

Fig. 3 10% Nb + V
TRANSFORMED AT 3000°C
PERMANENT SET VS. TIME

WASHINGTON AERONAUTICS DIV.
FIGURE 3
UNCLASSIFIED