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METALLURGY AND CERAMICS

UNITED STATES ATOMIC ENERGY COMMISSION

RESUME OF URANIUM ALLOY DATA

By
B. Lustman

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February 19, 1954

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For The Atomic Energy Commission

H. B. Canale

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B. Lustman

February 19, 1954

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Uranium Alloy Data for February 25 MeetingBy B. Lustman

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INTRODUCTION

Summarized below are additional data on uranium alloys which have been compiled at Bettis since the previous Panel Meeting. Of greatest significance probably are the results of preliminary irradiation tests on the U-Mo alloys and the results of cathodic treatment of clad and unclad samples of U-Mo alloys. Irradiation of an 11.5% Mo-U alloy to approximately 3000 MDW/T burn-up revealed no change in the general corrosion resistance of the alloy on subsequent corrosion testing but a marked decrease in the time for discontinuous failure. Cathodic treatment of unclad U-Mo alloys in boiling water revealed formation of uranium hydride needles in the alloy matrix. The time for discontinuous failure of samples thus treated on subsequent high-temperature corrosion testing is drastically reduced over that of untreated samples. Cathodic treatment of "defected" clad samples, however, revealed no pick-up of hydrogen in the form of hydride needles.

As a working hypothesis, it is suggested therefore that discontinuous failure is associated with the formation of a hydride phase in the alloy matrix, and that the effect of cladding in greatly prolonging the corrosion life of U-Mo alloys is a pressure effect operating by reducing the solubility of hydrogen in the metal. It is consequently believed that the accelerated discontinuous failure of the irradiated samples results from ionization of the water or free radical formation in the water by their highly energetic radiations and a consequent rapid pick-up of hydrogen by the metal. It is further believed that the cladding will be equally effective in preventing hydrogen pick-up (and thus discontinuous failure) in the case of ionized or atomic hydrogen produced by irradiation as was observed in the case of ionized hydrogen produced by cathodic treatment. It is highly encouraging that fission product contamination does not appear to accelerate the general corrosion rate of the alloy.

I. Mechanism of Corrosion of U-Mo AlloysA. Effect of Pressure on Corrosion Rate of U-Mo Alloys

As was pointed out in WAPD-MM-271, observations on clad fuel materials indicate a possible pressure effect on corrosion resistance, in that the time for mechanical disintegration of the alloy is greatly lengthened by containing it in a "defected" cladding. Comparison of specimens annealed at atmospheric pressure and at 5000 psi pressure at 500°C reveals that in an 8 w/o

Mo-U alloy the γ - α transformation is accelerated with increased pressure; since α formation is accompanied by decrease in general corrosion resistance, the effect of cladding restraint is not therefore believed to be an effect on transformation rate.

The effect of pressure on the initial corrosion rate has been determined and the results are shown in Fig. 1. These results were obtained in argon-steam atmospheres having 100 psi partial pressure steam at 650°F (343°C) and varying total pressures. Varying the partial pressure of steam did not alter the results. It may be noted that the corrosion rate is markedly pressure dependent and appears to decrease discontinuously at total pressures above about 2500 psi. Micrographs of the corrosion specimens reveal a surface layer containing a needle-like structure (presumably hydride) which appears to diminish in quantity and in thickness with increasing pressure. Figs. 2 and 3 represent the structures of two specimens similarly treated at 1,000 and 2,600 psi total pressures respectively. The presence of needlelike structures in the former and its absence in the latter are particularly to be noted.

B. Cathodic Treatment of U-Mo Alloys

Since UH_3 was believed to be a factor in the corrosion cracking of gamma uranium-molybdenum alloys, UH_3 was prepared electrolytically in these alloys. For this purpose various gamma uranium-molybdenum alloys were made cathode in a solution 0.1 N in total phosphate solution both at room temperature and at the reflux temperature of this solution. This solution, which served mainly as a carrier electrolyte, was buffered to pH 7.0 by dissolving the appropriate amounts of Na_2HPO_4 and NaH_2PO_4 . All specimens were cathodically treated for 7-9 days.

Platinum, which served as the anode, was also used for an electrical contact to the alloys studied. In some of the cases, the platinum wire dipped well below the surface of the phosphate solution, especially in boiling water tests. Under such conditions a large percentage of the hydrogen would be evolved at the platinum wire dipping below the solution as cathode. These cases are pointed out in Table I below. Also the alloy composition, heat treatment of the alloy, and current density of the sample as cathode are listed.

Selected photomicrographs are shown in Figs. 4 and 5. Fig. 4 shows the formation of profuse needles in a γ U-9.7% Mo matrix. No needles are formed in the interior of a U-9.7% Mo alloy (Fig. 5) which has been γ -quenched and aged for four weeks at 400°C; in this case, a separate layer forms at the surface. Note that four weeks at 400°C seriously reduces the high-temperature water corrosion resistance of this alloy composition.

X-ray examination of the sample in Fig. 4 revealed tentatively the presence of uranium hydride; examination of the sample in Fig. 5 showed definitely the presence of uranium hydride. The latter sample showed pits after 7 days and a high corrosion rate (0.9 mg/cm²-hr) on testing in water at 575°F, and disintegrated in 14 days. The former sample cracked in 7 days and disintegrated in 14 days. Note that samples of this composition as γ -quenched and not previously treated cathodically survive 30-50 days of corrosion testing prior to cracking.

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The corrosion of U-Mo alloys is therefore postulated to proceed according to the following scheme: transformed γ alloys corrode by the primary or concurrent formation of UH_3 and its subsequent oxidation by water at a correspondingly high general corrosion rate; alloys of the γ structure corrode primarily by the formation of the oxide with a correspondingly low general corrosion rate; release of nascent hydrogen by reaction and passage of hydrogen into the alloy results in the formation of hydride needles which eventually crack the material and lead to its mechanical disintegration.

C. Cathodic Treatment of Clad U-Mo Alloys

In order to study the effect of a Zircaloy-2 cladding on the formation of uranium hydride in a 10.5 w/o U-Mo alloy, both clad and unclad cylindrical specimens were drilled to the same depth to expose identical surfaces of the alloy; the bottom of each hole was approximately 1/8" in diameter. The rest of the surfaces were then coated with Glyptal and pressure sensitive mask-off type. Both specimens were then made cathode in boiling phosphate solution at a current density of 0.8 amps/cm² for 3 days. The specimens were then sectioned and examined metallographically. A drawing of the specimen sections and location of the photomicrographs is shown in Fig. 6. Photomicrographs at the selected locations are shown for the clad sample in Figs. 7, 8, and 9 and for the unclad sample in Figs. 10, 11, and 12.

Note that in the clad alloy, no trace of hydride formation is noted except at one location (Fig. 8) where a slight projection formed on drilling the defect. However in case of the unclad alloy, profuse hydride needle formation may be noted.

From these data as well as those discussed in section IA, the effect of cladding is postulated as follows. Since UH_3 has about half the density of uranium, the formation of vanishingly small amounts of this phase will result in sufficient volume dilatation to place the alloy meat of clad samples under considerable compressive stress. If it is further postulated that (as the results shown in Fig. 1 appear to indicate) the solubility of hydrogen and the formation of a separate hydride phase is severely reduced by applying pressures over 2500 psi to the alloy, and if, as indicated in section IB, the formation of a separate hydride phase is accompanied by mechanical disintegration, enclosure of the alloy in a cladding should, on the basis of these presumptions, result in the observed increase in corrosion life. Note that, in support of these conclusions, the burr of Fig. 8, at which location one would not expect cladding restraint to be effective, hydrided completely.

II. Alloy Development

A. Corrosion Resistance of Gamma-Quenched and Annealed Alloys

The major effort since the last Panel Meeting has been devoted to determining corrosion rates of various uranium-molybdenum base ternary alloys. The results are listed in Table II. These may be summarized as follows:

1. Ternary alloys containing Nb appear to show an optimum beneficial effect both on corrosion rate and thermal stability at about 1.5% Nb.

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2. Ternary alloys containing copper have as yet shown no effect; corrosion testing is continuing.
3. Ternary alloys containing silicon have as yet shown no effect; corrosion testing is continuing.
4. Ternary alloys containing aluminum show inferior corrosion and stability properties.
5. Ternary additions of Ti, Cr, and V show promise of improvement, at least in the as-quenched condition.

Testing is continuing on niobium and silicon additions to uranium. The former appear to be inferior to molybdenum alloys except at very high contents of niobium. The silicon alloys are promising, although their general corrosion rates are quite high. The relative corrosion resistances of the three classes of alloys are compared at various temperatures in Fig. 13.

The results of Table II may be compared with those for binary U-Mo alloys shown in Table III. It is evident that with respect to a combination of thermal stability and corrosion resistance, none of the ternary alloys equals the 12% Mo-U alloy.

B. Corrosion of Clad U-Mo Alloys

To confirm the effect of cladding in extending the corrosion life of uranium alloys, a number of "defected" clad samples are being tested. No data have been gathered thus far which contradict the observation made in WAPD-MM-271; however in most cases the testing times accumulated are too short to permit definite conclusions to be drawn.

The "defected" sample which had survived 167 days at 650°F was sectioned and examined metallographically. As may be noted from Fig. 14, the alloy had corroded away uniformly at the base of the defect. No trace of corrosion at the bond, of cracking, or hydride formation was evident at the base of the defect. The hardness of the metal had increased from 350 DPH to about 540 DPH. In spite of this hardness increase, no marked changes from the original gamma structure were noted. Samples cut adjacent to the defect when exposed to water bare had the same corrosion life (7-14 days) as samples of the as-extruded metal without the 167 day exposure at 650°F.

C. Transformation Kinetics

The mode and kinetics of transformation in U-Mo and U-Nb alloys are being determined. A T-T-T curve for a 5.4 w/o Mo-U alloy is shown in Fig. 15. The transformation product in the range 400-600°C is initially a finely-lamellar structure at the gamma grain boundaries which eventually consumes all the matrix (at the dotted line labelled 100% transformed). At still longer times (e.g. 250 hours at 550°C) the structure appears to coarsen; it is not known whether this denotes spheroidization or some other type of structural change. At 300°C, there appears to be a change in the mode of transformation; at 160 hours, the product which forms shows a faintly re-solvable needle-like structure.

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The mode of transformation in a 3% Nb-U alloy has also been observed. Such alloys show as-quenched a "distorted" alpha structure; the x-ray diffraction pattern exhibits a broad peak overlapping the positions of three peaks in completely annealed alpha-phase alloys. The microstructure for this treatment is shown in Fig. 16 and consists of a faintly resolvable needle-like structure within the gamma grains. On annealing at 400°C for various times up to 168 hours, a nodular transformation product forms at the gamma grain boundaries and at inclusions within the metal (Fig. 17) and eventually consumes the entire matrix (Fig. 18). Concurrent with this metallographic change, the alpha peaks become ever more clearly resolved. The results are summarized in Table IV.

The transformation mode in U-Mo alloys of high molybdenum content has also been noted. In Fig. 19 is shown the structure of 9.5% molybdenum alloy after 2 weeks annealing at 500°C. The structure shows a typically nodular transformation product and from x-ray diffraction is estimated to consist of 70% epsilon and 30% alpha. The structure of a nominally 13.5% molybdenum alloy similarly treated and shown in Fig. 20, is seen to exhibit a martensitic-like appearance and is estimated to consist of 90% and 10% α . The crystal structure of the epsilon phase is not certain. It has been reported by Pfeil in England to consist of an ordered face-centred tetragonal lattice of composition U_3Mo . Preliminary results from Bettis indicate the composition to be closer to $U_{11}Mo_5$ and the structure to consist of 16 atoms per unit cell.

III. Irradiation experiments on 11.5% U-Mo alloy

Post-irradiation experiments have been completed on seven natural uranium-11.5% molybdenum alloys irradiated in MTR for six weeks at estimated integrated fluxes of 1.5×10^{20} to 16×10^{20} neutrons/cm² (300-3000 MWD/T). The results of this experiment are summarized below:

Density - No changes were observed within experimental limits.

Hardness - A maximum increase of 36 DPH numbers was observed. The hardness increase was proportional to burnup.

Microstructure - Metallographic examination of samples from high flux and low flux regions showed no changes from the pre-irradiation gamma structure.

Dimensions - No changes in dimensions or shape were noted within the limits of experimental accuracy.

Electrical Resistivity - A decrease in electrical resistivity of about 2% was noted. The decrease was greatest in the samples exposed in low flux regions.

Corrosion Resistance - Non-irradiated control samples annealed 6 weeks at temperatures up to 400°C showed a general corrosion rate of 0.05 to 0.1 mg/cm²/hr at 575°C and most of the samples have survived over 60 days before mechanical disintegration occurred. The results of the post-irradiation corrosion tests are shown in Table V. These results may be summarized as follows:

2. The time for mechanical disintegration was drastically reduced from something over 60 days for the controls to 1 to 7 days in case of the samples receiving the maximum irradiations.

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TABLE I

Tabulation of Data on Hydrided Specimens

<u>Fig. No.</u>	<u>Alloy</u>	<u>Heat Treatment</u>	<u>Platinum Lead Immersed</u>	<u>Temperature of Test</u>	<u>C.D. (ma/cm²)</u>	<u>Interior</u>
4	U-9.7 w/o Mo	Gamma Quenched	No	100°C	15	P
5	U-9.7 w/o Mo	Gamma Quenched plus 4 weeks at 400°C	No	100°C	15	N
-	U-9.7 w/o Mo	Gamma Quenched plus 4 weeks at 400°C	No	100°C	15	N
-	U-9.7 w/o Mo	Gamma Quenched	No	25°C	50	S
-	U-6 w/o Mo	Gamma Quenched	Yes	25°C	50	N
-	U-6 w/o Mo	Gamma Quenched	Yes	100°C	15	S
-	U-12 w/o Mo	As Cast	Yes	100°C	15	N

N - No hydride needles in interior of sample
 S - Few hydride needles in interior of sample
 P - Many hydride needles in interior of sample

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TABLE II

Time in days for discontinuous failure in 650°F degassed H₂O

Alloy	900°C/24 hrs/W.Q.	900°C/24 hrs/W.Q. 400°C/1 week	900°C/24 hrs/W.Q. 400°C/3 weeks	900°C/24 hrs/W.Q. 400°C/5 weeks
6 Nb	4-7	0-1	0-1	
8 Nb	1-7	0-1	0-1	
10 Nb	14-28	0-1	0-1	
12 Nb	14-28	0-1	0-1	
16 Nb	28-42	28-42	0-1	
20 Nb	>70	>42 (end cracked)	14-28	
2 Mo 2 Nb	1-2	0-1	0-1	
4 Mo 4 Nb	7-14	0-1	0-1	
6 Mo 1.5 Nb	7-14	0-1	0-1	0-1
6 Mo 3 Nb	42-56	5-7	0-1	0-1
6 Mo 6 Nb	14-28	0-1	0-1	
7.5 Mo 1.5 Nb	>42	>28	>14	0-1
7.5 Mo 3 Nb	28-42	14-28	7-14	0-1
8 Mo 8 Nb	42-56	0-1	0-1	
9 Mo 1.4 Nb	>168 (end cracked off)	14-28	1-7	0-1
9 Mo 3 Nb	7-14	14-28	1-7	0-1
10 Mo 10 Nb	7-14	0-1	0-1	
12 Mo 1.5 Nb	14-28	>28	>14	>14
12 Mo 3 Nb	7-14	7-14	1-7	0-1
12 Mo 0.5 Cu	7-14			
12 Mo 1 Cu	>14	>7		
12 Mo 3 Cu	>14	7		
12 Mo 0.5 Si	>14	7		
12 Mo 1 Si	>14	7		
12 Mo 0.5 Al	1-7	1-2		
12 Mo 1 Al	1-7	0-1		
8.3 Mo .3 Cr	84-98	1-7	0-1	
8.3 Mo .6 V	84-98	1-7	7-14	0-1
8.3 Mo .3 Ti	>154	1-7	0-1	
3.8 Si	>28			
4.0 Si	14-28 (no discontinuous failure)			

TABLE III

Time in Days for Discontinuous Failure in 650°F Degassed Water in Binary U-Mo Alloys

Alloy	900°C/24 hrs/W.Q.	900°C/24 hrs/W.Q. + 400°C/1 week	900°C/24 hrs/W.Q. + 400°C/3 weeks	900°C/24 hrs/W.Q. + 400°C/5 weeks	900°C/24 hrs/W.Q. + 550°C/3 weeks
6 Mo	0-1	0-1	---	---	
9 Mo	14-28	1-7	0-1	---	0-1 (A + E)
10.5 Mo	28-42	42-56	14-28	7-14	---
11 Mo	28-42	14-28	14-28	28-42	---
12 Mo	42-56	42-56	42-56	56-70 (pieces still survive at 84 days)	14-28 (E)
15 Mo	14-28	14-28	7-14	14-28	1-7 (E + Mo)
18 Mo	1-7	1-7	1-7	1-7	---

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TABLE IV

Structures and Hardnesses of 3% Nb-U Alloys Aged at 400°C

	Heat Treatment		Hardness VHN - 1 Kg	X-ray Diffraction	Metallography
	Step 1	Step 2			
A	900°C, 24 hrs Water quench breaking bulb	-----	349	"Distorted α " Peak where 3 α lines should appear	Needle like structure
B	900°C, 24 hrs Water quench without break- ing bulb	-----	401	Not significantly different from "A" treatment	Same as "A" treatment
	Same as "B"	400°C, 4 hrs Water quench in bulb	502	Broad peak partially resolved into α peaks	Needles and some 2nd phase
	Same as "B"	400°C, 8 hrs	481	Progresses ↓	Progresses ↓
	Same as "B"	400°C, 16 hrs	485		
	Same as "B"	400°C, 24 hrs	491		
	Same as "B"	400°C, 168 hrs	475	α peaks fairly well resolved	Appears to be all 2nd phase

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TABLE V

Corrosion Rate of Irradiated 12 w/o Mo-U Samples in 575°F Water

Rate of weight loss in $\text{mg}/\text{cm}^2\text{-hr}$
Numbers in parentheses are weight losses in mg/cm^2

<u>Sample No.</u>	<u>Approximate Integrated Flux - neutrons/cm^2</u>	<u>1</u>	<u>7</u>	<u>14</u>
81	1.5×10^{20}	.05 (1.2)	.04 (6)	.04 (12)
40	4.4×10^{20}	.05 (1.2)	.03 (1.2)	Disintegrated
80	9.1×10^{20}	.05 (1.3)	Disintegrated	-
51	13×10^{20}	.04 (.9)	Disintegrated	-
90	16×10^{20}	.04 (.8)	Disintegrated	-
00	12×10^{20}	.05 (1.0)	1.88* (315)	1.72** (580)
61	5.8×10^{20}	.01	.01 (2)	.25* (83)

* Sample broke into two pieces

** Rate given is for the larger of the two pieces. The smaller sample disintegrated between 7 and 14 days.

Corrosion Rate vs. Pressure
with 100 psi Partial Pressure of Steam

Fig. 1

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Fig. 2 - U-8% Mo, Gamma Quenched. Exposed to Atmosphere of 900 psi Argon + 100 psi Steam at 343°C for 20 Hours.

250X

6740A-1

Fig. 3 - U-8% Mo, Gamma Quenched, Exposed to Atmosphere of 2500 psi Argon + 100 psi Steam at 343°C for 20 Hours.

250X

6519A-1

Fig. 4 - U-9.7% Mo, Gamma Quenched. Cathodically
Hydrided at 100°C. Cross Section
100X

6598A-1

Fig. 5 - U-9.7% Mo, Gamma Quenched. Aged 400°C,
4 Weeks, Cathodically Hydrided at 100°C.
Cross Section

100X

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Cross Section of Unclad Cathode

Cross Section of Clad Cathode

Fig. 6

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Fig. 7 - Clad sample, Section A of Fig. 6

Fig. 8 - Clad sample, Section B of Fig. 6

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Fig. 9 - Clad sample, Section C of Fig. 6

Fig. 10 - Bare sample, Section A of Fig. 6

Fig. 11 - Bare sample, Section B of Fig. 6

Fig. 12 - Bare sample, Section C of Fig. 6

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Corrosion Rates of Various Uranium-Base Alloys

Fig. 13

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Fig. 14 - Appearance of defect in 8.9% Mo-U alloy clad with Zircaloy after 167 days in 650°F water.

40X

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Fig. 16 - 3% Nb-U - 900°C, 24 hours
Water quenched

250X

Fig. 17 - 3% Nb-U - 900°C, 24 hours
Water quenched
400°C 8 hours
Water quenched

310X

Fig. 18 - 3% Nb-U - 900°C, 24 hours
Water quenched
400°C, 24 hours
Water quenched

250X

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Fig. 19 - 9.5% Mo alloy annealed 2 weeks at 500°C
Polarized light 250X

Fig. 20 - 13.5% Mo alloy annealed 2 weeks at 500°C
Polarized light 250X

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