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EFFECTS OF IRRADIATION ON SOME
CORROSION-RESISTANT FUEL ALLOYS

by

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EFFECTS OF IRRADIATION ON SOME CORROSION-RESISTANT FUEL ALLOYS

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ABSTRACT

An investigation was made of the behavior under irradiation of uranium-rich corrosion-resistant alloys with the following nominal compositions: U-3 w/o Nb, U-3 w/o Nb-0.5 w/o Sn, U_3Si (U-3.8 w/o Si), and U-2 w/o Zr (diffusion heat treated). The U-3 w/o Nb alloy in the rolled and gamma-quenched condition was highly unstable dimensionally under irradiation. The U-3 w/o Nb-0.5 w/o Sn alloy, in the cast and gamma-quenched condition, was only moderately stable dimensionally. The U_3Si in the cast condition showed good dimensional stability but in the extruded condition developed moderate anisotropic growth. Clad rods of diffusion heat treated U-2 w/o Zr alloy were generally highly stable. Only the U_3Si and U-2 w/o Zr alloy specimens retained a significant degree of their pre-irradiation corrosion resistance.

INTRODUCTION

A major engineering activity at Argonne National Laboratory has been concerned with the development of new and improved water-cooled reactors. In support of this effort, considerable experimental work is in progress to develop fuel materials which will permit higher performance of fuel elements in water-cooled reactors. The fuel development program is based on materials which show good corrosion resistance in water above 250°C. Both ceramic and metallic fuels are under study.

After a proposed fuel material has been shown to possess adequate corrosion resistance, mechanical properties, and fabricability, it is subjected to irradiation tests. Of particular interest are the dimensional stability of the material under irradiation and its ability to retain corrosion resistance during and after irradiation.

The results of initial irradiation experiments on various promising ceramic fuels have already been published.⁽¹⁾ The present report describes results of irradiation experiments on corrosion-resistant uranium alloys with the following nominal compositions: U-3 w/o Nb, U-3 w/o Nb-0.5 w/o Sn, U_3Si (U-3.8 w/o Si), and U-2 w/o Zr. The effects of

irradiation on the alloy U-5 w/o Zr-1.5 w/o Nb, the alloy chosen for the first core of the Experimental Boiling Water Reactor (EBWR), will be described in a separate report.⁽²⁾

Irradiations of the alloys were made in the MTR during the period 1954-1956.

SPECIMEN MATERIALS AND EXPERIMENTAL PROCEDURE

U-3 w/o Nb and U-3 w/o Nb-0.5 w/o Sn Alloys:

The selection of the U-3 w/o Nb and U-3 w/o Nb-0.5 w/o Sn alloys for irradiation tests was based on a study of corrosion-resistant uranium alloys containing niobium, tin, and zirconium.⁽³⁾ These alloys are most corrosion resistant when quenched from the gamma phase to form a martensitic alpha structure. Additional corrosion resistance can be obtained by a short aging heat treatment of the quenched material.

Specimens of the U-Nb alloy and the U-Nb-Sn alloy were supplied by the Foundry and Fabrication Group. The U-Nb alloy was rolled at 850°C and swaged to size. It was then heat treated by holding at 1000°C under vacuum for one hour, followed by quenching in Wood's metal. The U-Nb-Sn alloy was cast to size and was then given a heat treatment consisting of heating in lead at 800°C for four minutes, water quenching, holding at 350°C under vacuum for six hours, and air cooling.

U₃Si (U-3.8 w/o Si) Alloy:

U₃Si is the epsilon phase in the uranium-silicon binary equilibrium diagram.⁽⁴⁾ The compound is thermodynamically stable up to 930°C, at which temperature it undergoes a peritectoid decomposition. Work at Nuclear Metals, Inc. had shown the epsilon phase to be corrosion resistant in hot water.⁽⁵⁾ Later investigations at other sites confirmed the initial work.^(6,7)

Irradiation specimens of 10% enriched U₃Si were cast to size by the Foundry and Fabrication Group. Additional specimens of unenriched alloy were supplied in both cast and extruded conditions by Nuclear Metals, Inc. The enriched castings were heat treated by a two-hour anneal at 825°C under vacuum, followed by air-cooling. The unenriched castings were heat treated by holding at 800°C for 66 hours. The remaining unenriched specimens were cut from rods of U₃Si co-extruded with a 30-mil cladding of Zircaloy-2. The cladding was removed from half of the extruded specimens before irradiation.

U-2 w/o Zr Alloy:

The U-2 w/o Zr alloy was chosen for irradiation studies on the basis of results obtained at Nuclear Metals, Inc. It had been shown that U-2 w/o Zr alloy, coextruded with Zircaloy-2 cladding, was very resistant to corrosion near clad defects if the composite had been given a diffusion heat treatment prior to corrosion testing.⁽⁸⁾ The diffusion heat treatment consisted of an extended anneal at a temperature above 850°C, and resulted in interdiffusion of uranium and zirconium at the clad-fuel interface.

The specimens used in the present irradiation study were supplied by Nuclear Metals, Inc. They were made from U-2 w/o Zr alloy, coextruded at 620°C, with Zircaloy-2 cladding. The specimens were then given a diffusion heat treatment consisting of a two-hour anneal at 1050°C, followed by air-cooling. In the cladding of some of the specimens, a 0.007-in. hole was drilled prior to heat treatment; other specimens were treated in this manner after heat treatment. Also, one specimen was irradiated after the cladding had been removed by machining.

Table I gives the analyzed chemical compositions of the various alloy ingots from which irradiation specimens were made. Tables II through IV summarize the heat treatments investigated for each of the alloys.

TABLE I

Compositions of Alloys Used for Irradiation Specimens

Ingot No.	Manufacturer	C, ppm	N, ppm	Nb, w/o	Si, w/o	Sn, w/o	Zr, w/o
H-187	ANL ^(a)	95	240	3.02	-	-	-
Y16F-121	ANL	65	10	3.05	-	0.49	-
UA42	NMI ^(b)	425	-	-	3.82	-	-
Y16F-123	ANL	24	59	-	3.73	-	-
UA111	NMI	640	-	-	3.90	-	-
UA155	NMI	150	-	-	3.87	-	-
UA192	NMI	250 ^(c)	-	-	-	-	2.22

(a)ANL - Argonne National Laboratory

(b)NMI - Nuclear Metals, Inc.

(c)Estimated

The irradiation specimens were cylinders with diameters ranging from approximately 0.2 to 0.3 in. and lengths from approximately 0.8 to 1.5 in. The specimens were irradiated in loosely fitting capsules containing NaK for heat transfer. Each capsule contained an aluminum-0.5 w/o cobalt alloy flux monitor. The Co⁶⁰ activation provided data for calculation of fuel burnup and irradiation temperatures. The irradiation technique was developed for MTR conditions and has been described previously.⁽⁷⁾

TABLE II

Effects of Irradiation on U-3 w/o Nb and U-3 w/o Nb-0.48 w/o Sn Alloy Specimens

Spec No.	Ingot No.	Nominal Composition	Method of Fabrication	Heat Treatment	Burnup, a/o	Irradiation Temp, °C		Length Change, %	Growth Rate, $G_i^{(a)}$	Average Diameter Change, %	Weight Change, mg
						Surface	Center				
AA-120	H-187	U-3 w/o Nb	Rolled at 850°C	1 hr at 1000°C, quenched in Wood's metal	0.091	135	145	44	400	- 7.8	0.4
AA-117	H-187	Same	Same	Same	0.13	145	155	29 (b)	200	-	-
AA-118	H-187	Same	Same	Same	0.13	155	160	33	220	-12.1	-1.7
AA-119	H-187	Same	Same	Same	0.14	130	140	38	230	- 5.2	-0.3
AA-121	H-187	Same	Same	Same	0.19	160	170	45 (b)	200	-	-
AA-122	H-187	Same	Same	Same	0.37	130	140	(c)	-	-	-
BB-9	Y16F-121	U-3 w/o Nb-0.5 w/o Sn	Cast	4 min at 800°C, water quenched, 4 hr at 350°C	0.17	180	230	7.5	43	- 3.2	-1.0
BB-6	Y16F-121	Same	Same	Same	0.28	220	330	9.2	31	- 3.6	-6.7
BB-7	Y16F-121	Same	Same	Same	0.35	250	340	11.4	31	- 2.3	-4.5
BB-8	Y16F-121	Same	Same	Same	0.40	310	380	9.2	22	- 3.3	-0.5
BB-4	Y16F-121	Same	Same	Same	0.45	220	280	13.9	29	- 1.0	0.4
BB-5	Y16F-121	Same	Same	Same	0.54	310	410	8.8	16	- 1.9	0.2

(a) G_i = microin/in. per fission/ 10^6 total atoms

(b) Specimen fractured into two pieces under irradiation

(c) Specimen fractured into two pieces under irradiation, one of which was not recovered

TABLE III

Effects of Irradiation on U_3Si (U-3.8 w/o Si) Alloy

Specimen No.	Zircaloy-2 Cladding Thickness in.	Ingot No.	Method of Fabrication	Heat Treatment	Burnup a/o	Irradiation Temp, °C		Length Change, %	Growth Rate, G_i (a)	Diameter Change, %	Weight Change, mg
						Surface	Center				
AB-111	None	UA42	Cast	66 hr at 800°C	0.09	130	200	-1.44	-16	2.88	-
AB-110	None	UA42	Cast	66 hr at 800°C	0.25	280	450	2.05	8.1	1.80	-
BB-2	None	Y16F-123	Cast	2 hr at 825°C	0.14	220	290	0.92	6.6	2.34	-0.6
BB-3	None	Y16F-123	Cast	2 hr at 825°C	0.35	510	700	-1.40	- 4.0	2.35	-2.6
BB-1	None	Y16F-123	Cast	2 hr at 825°C	0.45	650	890	3.18	7.0	32.5	35.8
CE-4-1	None	UA111	Extruded at 850°C	7 days at 700°C	0.067	90	100	0.77	11	1.25	-1.2
CE-3-1	None	UA111	Same	Same	0.085	120	140	0.72	8.5	1.50	-0.6
CE-1-1	None	UA155	Same	Same	0.10	140	150	2.92	29	0.70	0.7
CE-2-1	None	UA155	Same	Same	0.13	140	160	3.00	23	0.70	1.0
CE-4-2	0.030	UA111	Same	Same	0.067	100	110	2.78	41	-1.9	1.8
CE-3-2	0.030	UA111	Same	Same	0.085	130	150	2.99	35	-0.05	-1.6
CE-1-2	0.030	UA155	Same	Same	0.10	140	170	3.88	38	-0.23	1.7
CE-2-2	0.030	UA155	Same	Same	0.13	150	180	3.94	30	0.02	1.3

(a) G_i = microin./in. per fission/ 10^6 total atoms

TABLE IV

Effects of Irradiation on Extruded and Diffusion Heat Treated U-2 w/o Zr Alloy (a)

Specimen No.	Zircaloy-2 Cladding Thickness, in.	Ingot No.	Burnup, a/o	Irradiation Temp, °C		Length Change, %	Growth Rate, G_i (b)	Diameter Change, %	Weight Change, mg
				Surface	Center				
CH-1-A	none (c)	UA192	0.086	130	150	5.27	60	-0.87	2.0
CH-2-A	0.027	UA192	0.074	120	140	-1.50	-20	-0.92	1.2
CH-2-B	0.029	UA192	0.074	120	140	0.12	1.6	-1.02	2.7
CH-2-C	0.028	UA192	0.074	120	140	-0.06	-0.8	-1.02	3.0
CH-2-D	0.028	UA192	0.086	130	150	-1.66	-19	-0.81	4.0
CH-1-B	0.028(d)	UA192	0.093	140	160	0.0	0.0	-1.04	2.5
CH-1-C	0.029(d)	UA192	0.093	140	160	-0.02	-0.2	1.24	2.9
CH-2-E	0.029(e)	UA192	0.086	130	150	0.0	0.0	-1.08	1.5
CH-2-F	0.028(e)	UA192	0.086	130	150	0.05	0.6	-0.47	1.8

(a) All specimens were extruded at 620°C, followed by a 2-hour anneal at 1050°C

(b) G_i = microin./in. per fission/ 10^6 total atoms

(c) Cladding was removed after heat treatment

(d) Cladding was defected with a 0.007-in. dia hole before heat treatment

(e) Cladding was defected with a 0.007-in. dia hole after heat treatment

As mentioned earlier, the primary objective of the irradiations was to determine the ability of the materials to retain their pre-irradiation corrosion resistance and dimensions. The aqueous corrosion rates of the specimens were determined by S. Greenberg before irradiation at temperatures ranging from 260 to 350°C. Similar temperatures were used for those specimens given corrosion tests after irradiation. Results from the corrosion tests have been previously reported.(7,9)

The weights and dimensions of all specimens were measured to within 0.1 mg and 0.0001 in., respectively, before and after irradiation for determinations of dimensional stability.

RESULTS AND DISCUSSION

The conditions of irradiation and dimensional changes observed in the specimens are summarized in Tables II through IV. The derivation of the growth coefficient, G_i , is given in reference 7. For small elongations

$$G_i \approx \frac{\% \text{ length change}}{\text{a/o burnup}}$$

U-3 w/o Nb Alloy:

The U-3 w/o Nb alloy was the first to be studied, and typical irradiated specimens are shown in Figure 1. It can be noted that the specimens were highly unstable dimensionally under irradiation. Values of G_i , the

Neg. No.	14625	15433	15431	15432

Burnup, a/o	0	0.091	0.13	0.14
Spec. No.	-	AA-120	AA-118	AA-119

Figure 1. Effect of irradiation at 140 to 160°C on typical specimens of rolled and gamma-quenched U-3 w/o Nb alloy. Magnification 2X.

growth coefficient, are shown in Table I and ranged from 200 to 400. Accompanying the length increases were corresponding decreases in the diameters of the specimens. Of the six specimens irradiated, two warped severely, as shown in Figure 1, and three other specimens were observed to have fractured transversely during irradiation. The elongation of the specimens during irradiation is attributed to a preferred grain orientation remaining from the fabrication procedure.

Surface roughening was also evident in all specimens. This is normally indicative of a large grain size, and in this case grain growth presumably occurred during the one-hour anneal at 1000°C.

Postirradiation corrosion tests at 260 and 290°C showed that the alloy had lost its corrosion resistance, even at the lowest burnup.⁽⁷⁾

U-3 w/o Nb-0.5 w/o Sn Alloy:

Figure 2 shows typical specimens of this alloy after irradiation. These specimens were cast to size so that a more random grain orientation was present. This is reflected in the lower values of the growth coefficient as compared with those of the rolled binary alloy. Values of G_i for the cast ternary alloy ranged from 16 to 43. All specimens decreased in diameter, however, indicating that some preferred grain orientation was present.

Neg. No.	16424	17669	17672	17670

Burnup, a/o	0	0.17	0.28	0.40
Spec. No.	-	BB-9	BB-6	BB-8

Figure 2. Effect of irradiation at 230 to 380°C on typical cast and gamma-quenched specimens of U-3 w/o Nb-0.5 w/o Sn alloy. Magnification 2X.

Warping and surface roughening were much less than in the binary U-Nb alloy. As can be noted in Table II, specimens of the ternary alloy were given a gamma solution heat treatment of only four minutes at 800°C. This brief anneal would permit much less grain growth than would be expected during the one-hour 1000°C anneal used for the binary alloy.

Specimens BB-9 and BB-5 were subjected to corrosion tests after irradiation. The specimens were completely disintegrated after 2.9 irradiation. The specimens were completely disintegrated after 2.9 days in water at 260°C,⁽⁹⁾ indicating that irradiation had destroyed their pre-irradiation corrosion resistance.

U₃Si (U-3.8 w/o Si) Alloy:

Photographs of cast specimens are shown after irradiation in Figure 3. The specimens maintained comparatively smooth surfaces, although some surface cracking was evident on specimen No. AB-110 and a deeper crack appeared on specimen No. BB-1. Growth coefficients, as shown in Table III, ranged from -16 to 8.1.

Neg. No.	17661	18645	17662	17658

Burnup, a/o	0.14	0.25	0.35	0.45
Irrad. Temp., °C	290	450	700	890
Spec. No.	BB-2	AB-110	BB-3	BB-1

Figure 3. Effect of irradiation on cast U_3Si (U-3.8 w/o Si) alloy. The dimensions of the specimens were not greatly affected by irradiation with the exception of specimen No. BB-1. Magnification 2X.

Of greatest interest in the group of cast specimens is No. BB-1, which was irradiated at a calculated center temperature of 890°C and a surface temperature of 650°C. The specimen exhibited severe swelling, as shown by its large increase in diameter and an unexplained weight increase. When an attempt was made to section transversely the specimen for metallographic examination, the specimen fractured and the appearance of the largest fragment is shown in Figure 4.

The specimen was found to contain a large central void. A cinder-like fragment was in the cavity but was not attached to the specimen. The interior surface of the void was smooth. A subsequent metallographic examination of the wall surrounding the cavity showed only a few scattered pores.

In contrast, metallographic examinations of swelled U-1.6 w/o Zr alloy specimens with large central voids have shown the interior surface to be very irregular and the metal surrounding the void to be highly porous.⁽¹⁰⁾

Figure 4

Appearance of U_3Si specimen No. BB-1 after transverse fracturing. The specimen was found to have developed a large central void surrounded by dense, pore-free metal.

18164

6X

Corrosion tests in water at 260 and 290°C of some of the irradiated cast U_3Si specimens^(7,9) showed that the specimens retained a high degree of corrosion resistance. The samples eventually failed because of mechanical disintegration, rather than because of corrosive attack.

Figure 5 shows typical irradiated specimens made from extruded U_3Si . The longitudinal striations on the clad specimens resulted from fabrication. Surface smoothness of the unclad specimens was excellent. This observation is in contrast to results reported by Bleiberg and Jones,⁽¹¹⁾ who found cracking and surface roughening in a group of extruded U_3Si samples subjected to comparable burnups and irradiation temperatures.

As shown in Table III, values of G_i , the growth coefficient, ranged from 8.5 to 29 for unclad material and from 30 to 41 for the clad specimens. It is evident that extruded U_3Si has a preferred orientation that causes anisotropic growth to appear during irradiation. The growth coefficients in the unclad specimens were comparable to that found for uranium which had been rolled at 300°C and quenched from the beta phase.⁽¹²⁾

The higher apparent growth rate of the clad specimens may have resulted from bimetallic thermal stresses during irradiation. Specimen CE-2-2, shown in Figure 5, displays rounded ends observed in all the clad specimens.

Neg. No.	19961	19957	19962	19958

Burnup, a/o	0.069	0.12	0.069	0.12
Cladding thickness, in.	None	None	0.030	0.030
Spec. No.	CE-4-1	CE-2-1	CE-4-2	CE-2-2

Figure 5. Effect of irradiation at 100 to 160°C on typical extruded specimens of U_3Si (U-3.8 w/o Si) alloy. Magnification 2X.

Most of the extruded specimens were subjected to postirradiation aqueous corrosion tests at 260, 290 and 315°C.⁽⁹⁾ It was found that raising the water temperature from 290 to 315°C often caused the failure mechanism to change from mechanical cracking, with little corrosive attack, to a corrosive type of failure accompanied by hydrogen evolution.

The generally favorable irradiation behavior of U_3Si with regard to both dimensional stability and retention of corrosion resistance indicates that further study of the material is warranted.

U-2 w/o Zr Alloy:

The only unclad specimen, No. CH-1-A, is shown after irradiation in Figure 6. The specimen warped and elongated with a growth coefficient of 60. This growth rate happens to be identical to that previously observed in other U-2 w/o Zr alloy air-cooled from the gamma phase.⁽¹³⁾

A typical clad specimen is shown after irradiation in Figure 7. As shown in Table IV, while the dimensional changes in the clad specimens were somewhat erratic under irradiation, they were considerably smaller

than shown by the unclad specimen. As might be expected, the defects drilled into the cladding before irradiation had no apparent effect on dimensional stability.

20217 2X
Figure 6. Effects of 0.086 a/o burnup at 150°C on unclad extruded diffusion heat-treated uranium-2 w/o zirconium alloy (specimen No. CH-1-A).

20219 2X
Figure 7. Effects of 0.093 a/o burnup at 160°C on extruded diffusion heat-treated uranium-2 w/o zirconium alloy with a 0.029-in. thick Zircaloy-2 cladding (specimen No. CH-1-C).

Postirradiation aqueous corrosion tests were performed on two of the clad specimens: No. CH-1-C, which was defected with a 0.007-in. hole before heat treatment, and No. CH-2-F which was similarly defected after heat treatment.⁽⁹⁾ Specimen No. CH-1-C was tested for 2.6 days at 290°C and then for 9.0 days at 315°C. Specimen No. CH-2-F was tested for

2.6 days at 290°C and then for 9.2 days between 315 and 338°C. No apparent changes developed in the samples, nor were changes observed in similar corrosion tests on unirradiated control specimens. It can be concluded that the effectiveness of the diffusion heat treatment is not impaired by irradiation to at least 0.1 a/o burnup.

CONCLUSIONS

1. Rolled and gamma-quenched U-3 w/o Nb alloy is subject to severe surface roughening and anisotropic growth under irradiation and does not retain pre-irradiation corrosion resistance.
2. Cast and gamma-quenched U-3 w/o Nb-0.5 w/o Sn alloy shows improved dimensional stability under irradiation but does not retain pre-irradiation corrosion resistance.
3. Cast U₃Si (U-3.8 w/o Si) alloy is relatively stable dimensionally under irradiation and retains a large degree of pre-irradiation corrosion resistance.
4. Extruded U₃Si (U-3.8 w/o Si) alloy is subject to anisotropic growth under irradiation but largely retains its pre-irradiation corrosion resistance.
5. The corrosion resistance of U-2 w/o Zr alloy which has been co-extruded with Zircaloy-2 and diffusion heat treated is unaffected by burnups of at least 0.1 a/o.

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