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"PIN-CUSHION" IRRADIATION TESTS OF URANIUM-
CHROMIUM ALLOYS

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"PIN-CUSHION" IRRADIATION TESTS OF URANIUM-CHROMIUM ALLOYS

S. H. Paine and F. L. Brown

ABSTRACT

Irradiation tests on small pins of wrought and heat-treated U^{235} -Cr alloys mounted in special heat transfer blocks or "cushions" are described. Three compositions, estimated at 0.03, 0.07 and 0.22 w/o Cr, were tested in three metallurgical conditions: alpha-annealed, beta-quenched, and isothermally transformed. Radiation damage to the specimens having the first two heat treatments was very similar to that observed in unalloyed uranium of comparable history, and the isothermally treated specimens were similar to beta-annealed uranium in behavior. In spite of this essentially null result, which is associated with failure to obtain grain refinement in the specimens, there is evidence that under proper conditions chromium additions to uranium may be beneficial. (auth)

A. INTRODUCTION

The U-Cr system is of the simple eutectic type (860°C , 20 a/o Cr) with eutectoidal reactions based on decreasing solid solubility of Cr in γ , β and α U (752°C , 640°C).^(1,2) Gamma solubility is estimated roughly at 5 a/o,⁽²⁾ whereas solubility of Cr in the beta phase of U is limited to 1.5 to 1.7 a/o maximum.^(2,3) The beta phase may be retained by rapid quenching; transformation kinetics have been investigated at Knolls Atomic Power Laboratory.⁽³⁾

The KAPL work revealed that heat-treated wrought U-Cr alloys in the range 0.45 to 3.9 a/o Cr showed greater stability under thermal cycling than did beta-quenched uranium.^(4,5) Improvement seemed to be optimum for specimens of approximately 1.8 a/o Cr which had been transformed by an interrupted quench. Attenuation of surface roughening was comparable to that obtained by Zr additions, whereas elongation stability was somewhat better.⁽⁶⁾ Inasmuch as irradiation tests of U-Zr alloys were in progress at ANL when these discoveries were announced,⁽⁷⁾ it seemed desirable to check the thermal cycling results by comparative exposures in Argonne's CP-3' reactor. S. Untermyer, therefore, arranged for the procurement of

wrought U^{235} -Cr pin specimens fabricated at Battelle Memorial Institute and there heat treated according to KAPL specifications. These were mounted at ANL in the "pin-cushion" mounts used in the U-Zr survey work⁽⁸⁾ and were compared with similar pins of U-2 w/o Zr and of unalloyed uranium fabricated at ANL.

Preliminary results of this study have been published.⁽⁹⁾ Data from the final irradiations and a summary of findings during the investigation are presented herewith.

B. DISCUSSION OF RESULTS

It was intended that the U-Cr specimens should contain 0.3, 0.6, and 2.0 a/o Cr (0.07, 0.13 and 0.45 w/o), but melting losses made the estimated contents only 50% of these values. The alpha fabrication and subsequent heat treatment of the samples are detailed in Appendix I. Three pins of each composition were prepared for irradiation in the following conditions:

1. Rolled; annealed at 575°C and furnace cooled.
2. Rolled; heated at 725°C and water quenched.
3. Rolled; heated at 725°C and isothermally quenched to transform at temperatures of 500°C-525°C.

Each heat-treated group was mounted in a separate heat-transfer block or "cushion," together with comparison pins of beta-quenched U or U-2 w/o Zr. Pins were permitted to project a distance equal to their diameter. Length measurements were made. The appearances of the specimens were recorded photographically after three successive periods of irradiation in the Argonne National Laboratory CP-3' reactor. Experimental procedures have been reported in detail elsewhere,⁽⁸⁾ and the complete irradiation history is given in Appendix II. Typical photographs of the specimens and their mounts are shown in Appendix III.

Changes in Length

The elongation behavior of all specimens which could be measured is presented graphically in Fig. 1, based on data summarized in Table 1.

As anticipated, the alpha-annealed specimens elongated radically. After the first exposure they were found to be shattered (Figs. 2 and 3, Appendix III), apparently because of mechanical restriction in the capsule. The cushion in which they were mounted was therefore removed from test. The 0.22 w/o pin broke off nearly flush with the cushion after elongating approximately 50%. The loose piece (Fig. 3-a) seemed quite sound and exhibited a "nail-head" condition at its free end, which is probably a

Figure 1

Effect of Burnup on the Radiation Growth Behavior of Specimens.

Table 1

SUMMARY OF RADIATION GROWTH DATA

Nominal Composition, w/o	Metallurgical Condition	Per Cent Elongation at Indicated Exposure(1)		
		First Exposure	Second Exposure	Third Exposure
U-0.03 Cr	Water quenched from 725°C	2.1	4.6	3.1
U-0.07 Cr	Water quenched from 725°C	4.3	8.6	9.1
U-0.22 Cr	Water quenched from 725°C	5.9	7.8	9.0
U-0.03 Cr	Slow interrupted quench, 725-500°C	-2.7	0.8	- 2.8
U-0.07 Cr	Slow interrupted quench, 725-500°C	-1.1	2.2	3.1
U-0.22 Cr	Slow interrupted quench, 725-525°C	0	0.3	2.5
100 U	Water quenched from 725°C	4.3	(2)	(2)
100 U	Water quenched from 725°C	3.2	7.9	9.3
U-2.0 Zr	Water quenched from 725°C	7.5	11.6	12.5

(1) The successive exposures integrated 0.15, 0.28 and 0.45% total uranium burnup in the Cr alloy specimens, slightly lower in the U and U-Zr specimens.

(2) Specimen withdrawn from test.

geometry effect. The 0.03 w/o specimen, however, disintegrated into many small fragments, and the portion remaining in the pin-cushion indicated that the failure was due to brittleness. The 0.07 w/o Cr specimen also failed, but apparently did not spall as radically as the pin having the lowest chromium content. The degrees of damage had a rough inverse correlation with chromium content.

As can be seen in Fig. 1, the beta-quenched U-Cr specimens were approximately equivalent to beta-quenched unalloyed uranium in their behavior, except for the 0.03 w/o Cr specimen, which was somewhat better. On the average, therefore, chromium additions may tend to slightly improve the stability of beta-quenched uranium, whereas 2 w/o Zr has the opposite effect.

The isothermally transformed specimens were definitely better than the beta-quenched specimens, when considered as a group. Their behavior appeared erratic, however. They tended to shrink under initial irradiation and then began to elongate slowly.

Not much can be said with certainty concerning the effect that variation of chromium content has on elongation behavior. As has been mentioned, the 0.03 w/o Cr beta-quenched specimen showed some superiority over the 0.07 and 0.22 w/o Cr pins. Whether the benefit is real is called into question by other considerations. During heat treatment the specimen materials were quarter-inch bars contained in small steel pipes, and the quenching speeds must have been somewhat less than optimum under these conditions. Normally, one would expect critical beta quenching of U-Cr alloys in this range of composition to produce the same type of transformation which is found in unalloyed uranium.⁽³⁾ The 0.07 and 0.22 w/o Cr alloys apparently behaved normally, but it is possible that the 0.03 w/o Cr specimen suffered some transformation in the upper C region of its TTT curve and therefore behaved more nearly like the isothermally transformed specimens. Because of statistical fluctuations no relative comparisons may be made between the interrupted quench specimens on the basis of differing chromium contents.

Surface Roughening

Surface roughening of all the quenched U-Cr specimens was quite gross, as may be seen in the photographic records of Appendix III. This was an unexpected development because specimens in the range 0.4 to 0.9 w/o Cr had remained smooth during thermal cycling tests at KAPL, whereas beta-quenched uranium had roughened.⁽⁶⁾ Moreover, in later tests at KAPL, specimens in the range of 0.12 to 0.26 w/o Cr appeared reasonably smooth after much higher burnup than was achieved in the present test.^(10,11) Roughening of the 0.07 w/o and 0.22 w/o Cr beta-quenched specimens was somewhat worse than that seen on the isothermally quenched specimens. In the case of the 0.03 w/o Cr alloy the behavior seemed to be reversed.

Gross roughening may be attributed to large grain size. It may be presumed that coarsening could have occurred either during the preparatory heat treatment of the specimen materials or during irradiation. It has been estimated that the specimens did not exceed 150°C during irradiation except as thermal contact between pin and cushion may have been broken by elongation and distortion. A study of the photographs shows that this happened to the alpha-annealed U-Cr specimens (Fig. 2), but there is no evidence that the condition resulted in the same grossness of surface roughening as seen on the quenched specimens (see Fig. 3). Extreme roughening would tend to improve the heat-transfer contact if unaccompanied by elongation. These considerations seem to rule out the possibility that grain coarsening occurred by overheating during irradiation.

The work cited above^(10,3) indicates that grain coarsening in certain of the quenched specimens should be expected as a result of the heat treatments they received. The kinetic behavior of the low U-Cr alloys seems to encourage coarsening if transformation takes place by the martensitic mechanism.⁽¹⁰⁾ Thus, the beta-quenched specimens would tend to coarsen. Also, because of its low actual chromium content, the isothermally transformed 0.03 w/o Cr specimen probably could be added to this group.⁽³⁾ This consideration would not apply, of course, to the 0.07 and 0.22 w/o Cr specimens which were isothermally transformed. However, all the specimens undoubtedly suffered from a certain lag in quenching rate because of the steel capsules in which they were heat treated. The two specimens last mentioned probably illustrate the magnitude of this effect.

In support of this argument it can be noted that the isothermally quenched 0.03 w/o Cr specimen suffered a much greater degree of roughening than either of the other pins having the same heat treatment (see Figs. 6 and 7). Some difference should be expected because of spread in the alloy content itself, an effect which is probably illustrated by the beta-quenched specimens (Figs. 4 and 5). Here a difference in degree of roughness is seen, but it is not as outstanding as in the case of the isothermally transformed specimens.

C. CONCLUSIONS

Discussion may be summarized as follows:

1. The U-Cr specimens tested in this experiment were distinctly coarse grained, judging from the grossness of surface roughening which occurred during irradiation. This was probably due to a combination of low alloy content, non-critical quenching in heat treatment and, in some cases, mode of transformation.

2. Slight gradations observed in the degree of radiation damage do give some evidence that increasing chromium content may have a beneficial influence on the stability of uranium under irradiation. However, in the composition range and under the conditions of this test the conclusion could be made that beta-quenched uranium is as good as beta-quenched U-Cr and that isothermally transformed U-Cr shows essentially the same behavior as beta-annealed uranium.

ACKNOWLEDGMENTS

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APPENDIX I

METALLURGICAL HISTORY OF SPECIMENS

Metallurgical processing of the materials from which specimens were fabricated has been detailed in a communication from Battelle Memorial Institute.⁽¹²⁾

The alloys were prepared by arc melting 16- and 18-gram charges. The arc-melted buttons were remelted six times and subsequently cast into 0.250" diameter rods in an attempt to produce homogeneous ingots. Nominal compositions of the ingots were intended to be 0.3, 0.6 and 2.0 a/o Cr (0.07, 0.13 and 0.45 w/o). On the basis of preliminary work, the charge analyses were set at 0.12, 0.24 and 0.60 w/o Cr, excess being added because of anticipated melting losses. Because of accountability, the enriched specimens were not analyzed after casting. However, later chemical analysis of similar specimens prepared in an identical manner with normal uranium indicated that the composition of the alloys was probably somewhat lower than expected, and that these alloys may have contained as little as one-half of the intended chromium content.

The cast alloy rods were enclosed in an iron jacket and reduced in cross section from 0.250" to 0.125" by hot rolling at 650°C. All the jackets remained intact during the fabrication cycle. The pins were dejacketed and the ends cut off to produce a 0.125" diameter specimen, 2" in length. Specimens were identified by a code numbering system and were heat treated in closures containing an argon atmosphere (see Table 2). For the quenched specimens these closures were steel pipes of small diameter. They were specified to prevent oxidation or loss of the small specimens in the lead pots which were used for heating and interrupted quenching.

From the heat-treated material the smaller pins (1/16" x 3/16") were machined for mounting in the pin cushions. The mounts were 7/8" in diameter, and comparison pins of beta-quenched uranium and U-2 w/o Zr were mounted at the center of each (see Table 2 for identification of these).

Table 2

IDENTIFICATION, COMPOSITION AND HEAT TREATMENT OF WROUGHT URANIUM
ALLOY PIN SPECIMENS IRRADIATED IN CUSHIONS 9, 10 AND 11

Specimen Designation			Alloy Content, w/o			Thermal History After 650°C Rolling
BMI No.	ANL No.		Charge Analysis	Intended	Estimated	
	Cushion	Pin				
X1A-1	9	1-1	0.60	0.45	0.22	Annealed 2 hr at 575°C; furnace cooled(1)
X2A-1	9	2-1	0.24	0.13	0.07	Annealed 2 hr at 575°C; furnace cooled(1)
X3A-1	9	3-1	0.12	0.07	0.03	Annealed 2 hr at 575°C; furnace cooled(1)
	9	Center			0.00	Annealed 10 min at 725°C; water quenched
X1A-2	10	1-2	0.60	0.45	0.22	Annealed 20 min at 725°C; water quenched(2)
X2A-2	10	2-2	0.24	0.13	0.07	Annealed 20 min at 725°C; water quenched(2)
X3A-2	10	3-2	0.12	0.07	0.03	Annealed 20 min at 725°C; water quenched(2)
	10	Center			0.00	Annealed 10 min at 725°C; water quenched
X1A-3	11	1-3	0.60	0.45	0.22	Annealed 20 min at 725°C; held 75 min at 525°C(2)
X2A-3	11	2-3	0.24	0.13	0.07	Annealed 20 min at 725°C; held 18 min at 500°C
X3A-3	11	3-3	0.12	0.07	0.03	Annealed 20 min at 725°C; held 20 min at 500°C
	11	Center			2.0(3)	Annealed 10 min at 725°C; water quenched

⁽¹⁾Annealed in a Vycor tube.

⁽²⁾Enclosed in small steel pipes and heat treated in a lead pot.

⁽³⁾2 w/o Zr. All others Cr.

APPENDIX II.

IRRADIATION HISTORY OF SPECIMENS

Specimens were irradiated in the ANL CP-3' heavy water reactor which operated, under normal conditions, at 275 kw and a water temperature of 35°C. Assembly and handling of the specimen capsule have been described elsewhere.⁽⁸⁾

Table 3 lists the irradiation data, and gives computed per cent surface depletions of U^{235} based on the reported neutron fluxes. Although flux perturbations are thus ignored, the numbers provide a relative scheme which can be normalized to observed burnup analyses taken after the final irradiation.

Table 3

IRRADIATION DATA, COMPUTED SURFACE DEPLETIONS OF U^{235} ,
AND ASSIGNED AVERAGE BURNUP LEVELS

Irradiation Date		Cushion Numbers	CP-3' Request No.	Integrated Reactor Power (kwh)	Equivalent Irradiation Time at 275 kw (10^6 sec)	Estimated % Surface Depletion of U^{235} (1)	Cumulative % Depletion of U^{235}	Estimated % Average Uranium Burnup Accumulated(2)
In	Out							
11-29-51	3-9-52	9, 10, 11	5003	438, 311	5.74	0.59	0.59	0.15
7-16-52	10-27-52	10, 11	5214	385, 501	5.05	0.52	1.11	0.28
11-24-52	6-22-53	10, 11	5286	793, 515	10.40	0.68	1.79	0.45

(1)Based on fission cross section of 500 barns and a reported flux of 2.04×10^{12} nv, except for Request No. 5286, which was assigned a flux of 1.3×10^{12} nv.

(2)Based on Cs^{137} analyses (Table 4) and the cumulative estimates of surface depletions of U^{235} .

The three analyses listed in Table 4 were obtained by radiochemical determination of Cs^{137} content. They are consistent with analyses from a much larger group of U-Zr pin specimens already reported.⁽⁸⁾ On the basis of these data the average flux in the U-Cr specimens is seen to be only 25% of the reported unperturbed flux in the reactor thimbles. As might be expected, this is somewhat lower than the 32% average flux found in specimens mounted in $1\frac{1}{4}$ " cushions with greater spacing between them. It will be seen that the center pin in Cushion 10 integrated less burnup than the outer pins, another indication that the flux gradients in the vicinity of the pins were appreciable.

The total uranium burnup accumulated at the termination of the third exposure is taken to be 0.45%, an average of the three Cs^{137} determinations. No conversion to total atom burnup is made, inasmuch as the correction is negligible for the range of composition involved. The total uranium burnups accumulated at the end of the first and second exposures

are taken to be 0.15 and 0.28%, respectively (Table 3), for the U-Cr pins. Burnup in the comparison pins is estimated to be 0.14, 0.26 and 0.41% of total uranium for the three exposures, respectively.

Table 4

ANALYSES OF AVERAGE TOTAL URANIUM
BURNUPS IN PIN SPECIMENS

Cushion No.(1)	Pin No.	Estimated U Content (w/o)	Estimated % Surface Depletion of U ²³⁵	Analyzed Per Cent Total Uranium Burnup
10	2	99.93	1.79	0.47
10	C	100	1.79	0.421
11	2	99.93	1.79	0.45

(1) Cushions were 7/8" diameter.

APPENDIX III.

POST-IRRADIATION DATA

The data on per cent changes of specimen length after the various exposures are summarized in Table 1 of the main report.

The appearances of all specimens were recorded photographically after each exposure in the reactor. This record was at first made by profile shots similar to those shown in Fig. 2, then by shots taken in the ANL stereobinocular microscope at 5-X (Figs. 2, 4 and 6), and finally by the Bausch and Lomb stereobinocular camera (Figs. 3, 5, 7 and 8). Figures 2, 3 and 8-a show the pins from Cushion 9; Figs. 4 and 5 are of Cushion 10; and Figs. 6, 7 and 8-b show Cushion 11 and its pins.

Figure 2. Appearance of Cushion 9 after Irradiation to 0.15% Uranium Burnup. Center pin is beta-quenched uranium; others are alpha-annealed U-Cr alloys, as indicated.

Figure 3. Appearance of Alpha-Annealed U-Cr Pins after 0.15% Uranium Burnup.

a) 0.22 w/o Cr Pins

17995

15-X

b) 0.03 w/o Cr Pins

17997

34-X

Figure 4. Appearance of Cushion 10 after Irradiation to Uranium Burnups of a) 0.15%, b) 0.28%, and c) 0.45%. Center pin is beta-quenched uranium, others are beta-quenched U-Cr alloys, as indicated.

a)

0.07 w/o

0.22 w/o

U

0.03 w/o

b)

0.03 w/o

0.07 w/o

U

0.22 w/o

c)

0.03 w/o

U

0.22 w/o

0.07 w/o

12560, 13310, 14471

5-X

Figure 5. Appearance of Beta-Quenched U-Cr Pins after 0.45% Uranium Burnup.

a) 0.22 w/o Cr Pin

17998

34-X

b) 0.03 w/o Cr Pin

17999

34-X

Figure 6. Appearance of Cushion 11 after Irradiation to Uranium Burnups of a) 0.15%, b) 0.28%, and c) 0.45%. Center Pin is Beta-Quenched U-2 w/o Zr; others are isothermally transformed U-Cr Alloys, as indicated.

a)

0.03 w/o

0.22 w/o

2 w/o Zr

0.07 w/o

b)

0.07 w/o

0.03 w/o

2 w/o Zr

0.22 w/o

c)

0.03 w/o

0.22 w/o

2 w/o Zr

0.07 w/o

12559, 13311, 14472

5-X

Figure 7. Appearance of Isothermally Transformed
U-Cr Pins after 0.45% Uranium Burnup.

a) 0.22 w/o Cr Pin

18001

34-X

b) 0.03 w/o Cr Pin

18002

34-X

Figure 8. Appearance of Beta-Quenched Comparison Pins from Cushions 9 and 11 after 0.15% and 0.45% Uranium Burnups, Respectively.

a) Uranium Pin from Cushion 9

17994

34-X

b) U-2 w/o Zr Pin from Cushion 11

18000

34-X