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"PIN-CUSHION" IRRADIATION TESTS OF URANIUM
AND ITS ZIRCONIUM ALLOYS

by

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"PIN-CUSHION" IRRADIATION TESTS OF URANIUM AND ITS ZIRCONIUM ALLOYS

by
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ABSTRACT

An irradiation test of 1/16 in. diameter x 3/16 in. long U^{235} and U^{235} -Zr alloy specimens, in the range 2 to 20 w/o Zr, is described. The small pins were mounted in heat transfer blocks, or "cushions," in such a way that surface roughening and length changes could be observed, recorded, and compared for five stabilizing heat treatments up to exposure levels of approximately 1% total atom burnup. Addition of 2% Zr was found to be very beneficial, but the behavior of the alloys underwent distinct changes in going from 2 to 4 w/o Zr. Material, both as-cast and subsequently isothermally treated, changed from minus to plus elongation, whereas wrought materials, either gamma-slow cooled or isothermally treated, changed from plus to minus behavior. Beta-quenched wrought material varied unpredictably. Addition of Zr up to 20% showed further improvement in stability for all treatments. Factors which may have contributed to this behavior are discussed.

A. INTRODUCTION

The first studies of dimensional stability of uranium and its alloys were plagued with the problem of how to remove fission heat from specimens without imposing restraints which would destroy much of the usefulness of the experiment. Perhaps the simplest way to remove heat is by solid contact through a tightly compressed sandwich assembly around a fuel wafer. The first study of radiation damage at ANL was made with such an arrangement,^(1,2) and it was found that the specimens tended to stick to the sandwich pieces at relatively low exposures, making removal difficult. In addition, the specimens had to be quite thin, because of their enrichment, and the restraint imposed upon them obviously suppressed dimensional reactions which were trying to occur. For these reasons the method was abandoned.

It was then proposed that small pin specimens be embedded in heat-transfer blocks in such a way that a short portion of their length would project and be free from restraint. Thus the "pin-cushion" idea was developed and was used to investigate, in the ANL CP-3' reactor, the radiation behavior of a series of uranium-zirconium alloys. The arrangement

which was devised proved to be relatively easy to irradiate in a capsule and to examine periodically. Stereophotos could be taken to record relative surface appearance of the pins and the lengths of the specimens, and their mounts could be measured directly. This limited information, however, has permitted good qualitative deductions concerning radiation-damage trends in the U-Zr system.

Incomplete results from the pin-cushion tests have been reported elsewhere.^(3,4,5) All the final data on CP-3' exposures of uranium-zirconium alloys are given in the present report, including those from an irradiation of modified pin-cushions in liquid metal NaK. This latter experiment was made possible by the proved feasibility of using caustic metals as heat transfer agents in thermal reactor specimen capsules.⁽⁶⁾

B. EXPERIMENTAL PROCEDURE

1. Specimens and Their Irradiations

The fuel materials to be studied were supplied as small pins measuring approximately $3/16$ in. $\times$ $1/16$ in. diameter. As detailed in Appendix I, the pins were vacuum cast in thoria crucibles, jacketed in steel and rolled at 600°C , heat treated, machined to size and then stress relieved at 575°C . These pin specimens were mounted in upright positions in holes in disc-shaped mounting blocks; the metal around the base of each projecting pin was carefully pressed, or peened, to provide intimate contact. Three or four pins were thus mounted in each block - their appearance suggesting the name "pin-cushion." Two to four pin cushions were assembled in tandem in capsules for irradiation. Two capsule designs were used and, in each case, the capsule function was dual purpose: not only did the capsule provide protection for the test specimens, but it also protected the reactor test facility from possible contamination due to specimen rupture or disintegration.

Figure 1 illustrates the dry capsule and pin-cushion design used most extensively in the tests. The pins were embedded in zirconium cushions, and their exposed ends were protected by aluminum spacer-covers. Two-thirds of the length of each pin was embedded in the zirconium to provide heat removal by conduction through the base. The lifting knob on the top spacer was provided for extracting the capsule from the reactor.

The wet capsule used in later tests is shown in Fig. 2. The pins were of the same size as those used in the first test; however, the cushions were fabricated from aluminum and an assembly of four cushions was sealed in a NaK-filled steel capsule. As the NaK bath surrounding the pin cushions would provide efficient heat removal during irradiation, $2/3$ of the length of each pin was permitted to project from the cushion and $1/3$ of each pin was embedded in the aluminum cushion.

Figure 1. Dry Irradiation Capsule and Pin Cushion Assembly
(Dimensions in inches)

Care was exercised in mounting to assure no mixup of pins. A numbering system was devised and the proper identifying numbers of the pins were stamped on the cushions immediately adjacent to the pins. Cushions also were assigned identifying numbers, and, in the case of the dry capsules, the numbers were stamped on the outer rims, where they could be observed without disassembly.

The pin-cushion capsules were irradiated in the CP-3' reactor. Most of the irradiations were in the high-flux central thimble; however, two assemblies were irradiated in side thimbles due to lack of test space in the central thimble. Exposure periods extended over a span of about three years. The pins were examined periodically at first, but finally most of them were left in the reactor to accumulate a maximum of flux until exposures of other specimens in the MTR should overtake them. The pin specimens immersed in NaK received only one irradiation exposure because the NaK-loading device could not be used remotely to re-encapsulate the irradiated cushions. Detailed information regarding irradiation exposures and computations of burnup, etc., is given in Appendix II.

2. Handling of Capsules in Cave

The dry pin-cushion capsules were easily assembled by hand before the first irradiation exposure. Opening the capsules after each irradiation, however, proved to be quite difficult at times. The capsules were held upright in a vise and a nut driver was used to loosen the nuts on the lag bolts. Rather high torques were usually experienced; a lever arm arrangement on the nut drivers was required. Particular difficulty was experienced with the 7/8-in. diameter, bolted capsules, because the lifting knob interfered with the use of a nut driver and the nuts had been filed so that they did not project beyond the diameter of the capsule. Therefore, a small adjustable wrench, instead of the nut driver, had to be used on the small capsules. Separating the cushions and spacers also was difficult. Whenever an initial attempt to pry them apart with a chisel or screw driver before withdrawing the lag bolts was unsuccessful, the bolts were removed completely and the sections tapped with a hammer to loosen the joints. Great care was required to prevent accidental damage to the pins. Sometimes it was suspected that a pin might have disintegrated and the disassembly procedure was then carried out with the capsule submerged in carbon tetrachloride or acetone to prevent airborne contamination within the cave facility. Re-assembly of the dry capsules was not particularly troublesome.

No particular difficulties were experienced in opening the NaK-filled capsule. It was girdled with a slitting saw and the contained NaK dissolved in alcohol.

3. Examination Procedures

In general, photographic records and dimensional measurements were made before and after irradiation exposure. After the final exposure the accumulated burnup of U^{235} in selected pins was obtained by radio-chemical analysis for Cs^{137} .⁽⁷⁾ Actual details of the procedure were improved during progress of the experiment, and the changes are apparent in the illustrations appended.

Visual Examination and Photography

Optical equipment for visual observation and photography was greatly improved during the course of the experiment. In the early work visual examination was limited to what could be seen through a 2-foot window with the aid of a large magnifying glass. Photographic records were first made with a long focal length copying lens installed in the cave wall and permitting magnification of only 3X. Next, stereo viewing and stereophotography at 13X and 5X became possible through the development and construction of a periscope having a double optical system. The quality of the transmitted image was good; however, focusing was difficult due to poor light transmission and the field of view was limited. This ANL-developed stereo periscope was used for most of the examination and photographic work. Later, a commercially developed stereo periscope was procured (manufactured by Bausch and Lomb Optical Company) and stereo examination and photography at magnifications as high as 34X became possible without troublesome adjustments.

Figure 3 is a comparison of the photographic work obtained by the three above methods. Profile shots were made with the old camera to record the degree of surface roughening. Angle shots were made with the ANL and the B & L stereo periscopes. As shown in Fig. 3, the ANL version had a 1/2 in. x 5/8 in. field of view. The depth of focus was good, however, and by orienting the pin-cushion in the proper direction, all of the pins in a cushion could be included in the picture. The B & L stereo periscope brought considerable improvement in the field of view.

Measuring Procedure

Measurements of lengths of the pins were made before and after each irradiation exposure. The dimension actually measured was the distance from a base point on the cushion (the top or bottom of the cushion) to the top end of each pin. A micrometer was used in the first test but was discarded in favor of a dial gage which was more easily handled by remote manipulation. Even with the dial gage, however, measuring proved to be an arduous task because of difficulty in aligning the tiny pins under the dial gage stylus. Up to ten readings were made on each pin; clearly suspect values were ignored and average values were reported. Measurements made on zirconium

Figure 3. Examples of Photographic Results
Obtained by Methods Described in Text.

(a)

12587, 12582, 12583

3-X

(b)

14468

5-X

(c)

17982

4-X

- (a) Cushion 5, as seen by first ANL macro camera (0.3% U burnup)
 (b) As seen by ANL stereo binocular periscope (0.7% U burnup)
 (c) As seen by B & L stereo binocular periscope (1.0% U burnup)

control pins in many of the cushions attested the inherent accuracy of the method; however, apparent changes in length brought about by longitudinal warp or end roughening were not discerned as such.

During the first examinations an effort was made to measure the diameter of the pins. The procedure consisted in taking a plan-view photograph of a pin-cushion, measuring the diameters of fuel pins and the zirconium control pin on the photographic negative, and then normalizing the diameter values, using the known value for the diameter of the Zr control pin. Even though an optical comparator was used, it was extremely difficult to determine diameters precisely and the effort was abandoned.

Metallography

At the conclusion of irradiation exposures and examinations, a number of pin specimens were broken from their cushions for metallographic polishing and examination. The specimens proved too small to mount and polish remotely with success, however. The difficulties came to a climax during the final mechanical and electrolytic polishing stages, when staining of the prepared surfaces always occurred, completely obliterating the tiny cross sections and making microscopic examination impossible.

Chemical Sampling

One pin from each cushion was submitted to the ANL Chemical Engineering Division for uranium and fission product analysis.⁽⁷⁾ These specimens were taken by clamping the cushion in a vise, placing a small chisel against the pin and shearing the pin by a sharp hammer blow to the head of the chisel. The entire projecting portion of the pin thus became the chemical sample.

C. DISCUSSION OF RESULTS

Most of the observations included in this section are based on two classes of data: (1) measurements of pin lengths, and computations of the total atom burnups with which they were associated, and (2) photographs of the pins, whereby visual comparisons of radiation damage were made possible. Both classes of data yield only qualitative results, although the length measurements have been treated quantitatively with great care. Physical conditions to which the specimens were subjected during irradiation were not definable enough to justify pin-point deductions. For instance, in the dry capsule the temperature was a variable which could not reasonably be approximated for any specimen, due to uncertainties about conditions of heat transfer. In this case it seems reasonable to assume that specimens which showed the lowest elongations integrated their damage at lower temperatures than those which elongated enough to separate from their mounts (see Figs. 12, 16, 17, etc., Appendix III). Deductions obtainable from the data are influenced by such factors.

1. Changes in Length

The length data are summarized (from Appendices I and II) according to specimen composition and heat treatment in Tables 1 and 2.

TABLE 1

Burnup and Elongation Data for U and U-Zr Pin Specimens from Dry Capsule Irradiations.⁽¹⁾

Nominal Zr Content (w/o)	Fabrication and Heat Treatment ⁽²⁾	Total Atom Burnup (%)	Elongation (%)	Total Atom Burnup (%)	Elongation (%)	Total Atom Burnup (%)	Elongation (%)	Total Atom Burnup (%)	Elongation (%)
0	725°C quench, 575°C anneal	0.08	5.1	0.19	9.4	0.47	14.9	0.86	19.6
		0.07	3.5						
	725°C anneal 575°C anneal	0.07	9.7						
		0.07	15.5						
2	As cast 725°C quench, 575°C anneal	0.27	-0.6	0.29	1.6	0.69	-4.8	0.96	-2.5
		0.08	4.4						
4	As cast 725°C quench, 575°C anneal	0.28	13.9	0.66	13.5	0.91	15.8	0.96	10.1
		0.07	4.4						
		0.20	13.9						
		0.20	9.1						
6	As cast 725°C quench, 575°C anneal	0.13	4.8	0.62	9.3	0.87	9.2	0.40	8.1
		0.27	5.9						
10	As cast 725°C quench, 575°C anneal	0.12	2.8	0.56	6.7	0.78	5.9		
		0.13	25.0						
20	As cast 725°C quench, 575°C anneal	0.08	1.2	0.41	2.3	0.57	2.0	0.41	7.4

(1) Taken from Tables 9 and 10, Appendices II and III.

(2) All specimens rolled at 300°C before heat treating, unless otherwise stated.

TABLE 2

Burnup and Elongation Data for U and U-Zr Pin Specimens from Wet Capsule Irradiations.⁽¹⁾

Zr Content (w/o)	Fabrication	Heat Treatment	Total Atom Burnup (%)	Elongation (%)	Estimated % Elongation at 1% Burnup
0	Rolled at 300°C	725°C quench, 575°C anneal	0.28	4.8	17
2	As cast	None	0.27	-0.6	-1
4	As cast	None	0.25	8.8	12
6	As cast	None	0.24	7.2	10
2	As cast	800°-550°C isothermal quench	0.27	-4.6	-6
2	Rolled at 300°C	800°-550°C isothermal quench	0.27	7.2	7
2	Rolled at 300°C	800°-550°C isothermal quench	0.27	4.5	
4	Rolled at 300°C	800°-550°C isothermal quench	0.25	-4.3	-6
6	Rolled at 300°C	800°-550°C isothermal quench	0.24	-2.7	-4
2	Rolled at 300°C	800°C slow cool	0.27	5.9	7
4	Rolled at 300°C	800°C slow cool	0.25	-2.9	-4
6	Rolled at 300°C	800°C slow cool	0.24	-1.6	-2

(1) Taken from Tables 9 and 10, Appendices II and III.

Figure 4 is a chart which sets forth the relative behaviors of the different alloys and heat treatments, estimated at 1% total atom burnup from a

Figure 4. Dependence of Per Cent Elongation at 1% Burnup Upon Zirconium Content and Heat Treatment in U and U-Zr Pin Specimens

critical review of all the data. The results are not statistically strong, but none of the data for the five best heat treatments in the two tables have been ignored in this graph. The 1% burnup extrapolations from Table 1 were obtained by plotting the data as shown in Figs. 5 to 8. Anomalous points are indicated, as well as the more uniform trends. It will be noted that the beta-anneal and the alpha-anneal studies, graphed in Figs. 7 and 8, are not included in the summary represented by Fig. 4, inasmuch as these treatments are clearly deficient when compared with the others. The extrapolations to 1% burnup listed in Table 2 are admittedly tentative, inas-

much as the data on which they are based are single points at approximately 1/4% burnup. It has been assumed that the relative behaviors of the specimens can reasonably be projected to 1% burnup, and that saturation tendencies similar to those shown in Figs. 5 to 8 for the dry capsule specimens are also applicable.

A striking change in the elongation behavior of the various alloys is clearly seen between 2 and 4 w/o Zr. Three aspects of this change are noted:

a. Wrought specimens subsequently quenched from 725°C (beta quench) go from a fairly predictable modest elongation rate at 2% Zr to unpredictably large or small rates at 4% Zr.

b. Wrought specimens subsequently treated at 800°C and either isothermally quenched or slow cooled lengthen somewhat less than beta-quenched specimens when only 2% Zr is present, but they shrink when the alloy content is increased to 4%.

Figure 5. Effect of Total Burnup on Elongation of As-Cast U-Zr Pin Specimens.

Figure 6. Effect of Total Atom Burnup on Elongation of Beta-Quenched U and U-Zr Pin Specimens (Rolled at 300°C, 725°C quench, 575°C anneal)

Figure 7. Effect of Total Atom Burnup on Elongation of Beta-Annealed U and U-Zr Pin Specimens (Rolled at 300°C, 725°C anneal)

Figure 8. Effect of Total Atom Burnup on Elongation of Alpha-Annealed U and U-Zr Pin Specimens (Rolled at 300°C, 575°C anneal)

c. Cast specimens, as-cast or isothermally treated from 800°C subsequently, shrink if only 2% Zr is present. As-cast specimens lengthen when the amount is 4%.

The anomalous behavior of the specimens quenched from 725°C might be associated with the fact that this temperature equilibrates the 2%, 10% and 20% Zr alloys in two-phase regions.⁽⁸⁾ However, the 4% and 6% Zr alloys equilibrate as single-phase gamma at this temperature. In the specimens under study all compositions except 2% and 20% Zr have essentially the same erratic behavior. Therefore, it seems evident that other factors are involved.

Because of the small number of samples involved, the question arises whether the inversion effects, (b) and (c) above, are real. That the shrinkages are real is a matter of observation, but there is some probability that specimens could have been wrongly identified while mounting them in the cushions - in spite of the great care exercised in this operation (Appendix I). Because of the continuity of data from 4 to 20 w/o Zr the possibility of a significant mixup in all except the 2 w/o specimens may be dismissed. Of the five treatments shown in the figure, possibility of an error in identification of the as-cast and of the beta-quenched specimens may immediately be rejected with very small risk. This follows because: 1) the data for beta-quenched specimens are based on three tests, two of which did not involve any of the other four treatments of 2 w/o material, whereas one involved comparison with the as-cast treatment; 2) the data for as-cast specimens are from two tests, one of which was entirely independent from the other four treatments of 2 w/o material. There is a greater chance that the other three treatments may have lost their right identification because all of them were involved in a single test. However, neither of the two alternative arrangements of their identifications can remove both of the inversions which are involved.

It may reasonably be argued, therefore, that the change in growth behavior of the as-cast 2 w/o Zr material is real and that at least one of the other indicated inversions is also real. There seems to be no valid reason for not accepting all of them.

A cause for the behavior cannot be assigned with any assurance. Subsequent tests of different materials similarly heat treated, using a different wet experimental method, in a different reactor, have failed to reproduce it. Alpha-rolled materials sometimes show a shrinkage behavior when tested without further re-orienting heat treatment,⁽⁹⁾ but shrinkage of as-cast or of beta or gamma-treated material has not been observed.

Examination of the U-Zr phase diagram⁽⁸⁾ adds no clue of recognizable significance, although it is noted that the 2 w/o composition is close to the peritectoid shown in the reference.

2. Changes in Surface and Shape

The stereophotos in Figs. 10 to 23 (Appendix III) record the appearance of the pin specimens after irradiation. They provide the basis for the qualitative observations which follow.

The cast alloys tend to roughen and go out of shape worse than those which have been wrought and heat treated. In this respect, those irradiated dry appear worse than those which had heat removed by liquid metal contact (compare Figs. 14, 20, 22, 23). The one cast specimen (2 w/o Zr) which had the 800-550°C isothermal treatment before irradiation shows definite improvement over the as-cast specimens (compare Fig. 19b with Fig. 20f). This specimen also held its shape better than the comparable wrought and isothermally quenched specimen (compare Fig. 20e with Fig. 20f).

Specimens quenched from 725°C show the smoothest surfaces and generally retain their shapes better than do those having the other heat treatments, except when anomalous elongation is present (compare Fig. 12 with Fig. 15; also see Fig. 19c). The 2% Zr beta-quenched specimen in cushion 6, however, is one of the few specimens which showed extensive cracking (see Fig. 15b). The surface smoothness of specimens heat treated from 800°C tends to be intermediate between those cast and those wrought and quenched from 725°C.

The improvement in surface condition which 2% Zr gives to uranium (compare Fig. 18a with Figs. 18, 19; also Figs. 10, 11 with others) is noticeably increased by 4% Zr (compare Figs. 10, 14, 15; also Figs. 19, 20 with Fig. 22). The higher zirconium alloys, however, do not improve in proportion to their compositions, with the possible exception of the as-cast material (see Fig. 13).

Surface stability in specimens which have reasonably good elongation stability is very apparently associated with grain refinement. From an analogy with thermal cycling work⁽¹⁰⁾ it was suspected from the outset of the experiment that radiation surface stability in fuel metals based on alpha uranium might be associated with refinement of grain. That such is the case is now a matter of common knowledge⁽¹¹⁾ and the evidence in this series of irradiations is all compatible with this view.

D. SUMMARY

The findings may be summarized briefly, as follows:

1. Under the conditions of the test the addition of 2 to 20 w/o Zr to U is shown to have a beneficial effect on dimensional stability under irradiation, as measured by the anisotropic elongation behavior. The benefit, in general, appears to increase with zirconium content; there are two reservations to this statement, mentioned below (2 and 4).

2. The behavior of 725°C quenched specimens appears erratic. The most stable specimens continue a trend to improvement with zirconium content, but above 2 w/o Zr anomalous scatter occurs so that the stability of some specimens is worse than that of beta-quenched unalloyed uranium.

3. The 4 w/o Zr as-cast specimens are not quite as stable as the 2 w/o specimens, but from 4 to 20 w/o stability improves.

4. The following metallurgical treatments are consistently good:

- a. As-cast (Good over the whole range from 2-20 w/o Zr)
- b. Cast, 800-550°C isothermal treatment. (2 w/o Zr only)
- c. Alpha rolled, 800°C slow cool (2-6 w/o Zr)
- d. Alpha rolled, 800-550°C isothermal treatment. (2-6 w/o Zr)

5. An inversion of growth behavior from positive to negative (or vice versa) in the span from 2 to 4 w/o Zr is seen in specimens having three of the metallurgical treatments mentioned above; the experiment does not differentiate the cause of this phenomenon:

- a. As-cast changes from negative to positive
- b. Alpha-rolled, 800°C slow cool changes from positive to negative
- c. Alpha-rolled, 800-550°C isothermal treatment changes from positive to negative.

6. Visual appearance of the specimens is compatible with the thesis that surface stability is associated with grain refinement. As-cast surfaces were roughest, quenched surfaces smoothest, and those that had undergone modified or interrupted cooling were intermediate. Increasing Zr content from 2 to 4 w/o noticeably improved the surface stability for all treatments; further increases caused little, if any, added benefit except in the as-cast condition.

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APPENDIX I

Metallurgical History of U-Zr AlloysI. Development and Fabrication Procedures, Using Normal Uranium

It was desired to fabricate 1/16 in. x 3/16 in. wire specimens of 0, 2, 4, 6, 10 and 20 w/o Zr alloys. Some of these were to be of as-cast, some of wrought and beta-quenched, some of wrought and beta-annealed, and some of wrought and alpha-annealed material.

Since no information was available on the methods of casting and fabricating the low zirconium-uranium alloys, a series of experimental melts of the various alloys were made using normal uranium. The limited quantity of enriched uranium available for the job made it necessary to limit the size of casting to 30 or 40 grams.

a. Equipment Used: A vacuum melting furnace described elsewhere⁽¹²⁾ was modified to take a 4 in. diameter quartz tube. One of the water-cooled molds was converted into a cooling chamber by the installation of a movable zirconia crucible pedestal mounted on a water-cooled stainless steel rod, operated through a rubber gland in the bottom of the mold. This allowed a small crucible of molten metal to be withdrawn from the hot zone of the furnace into the water-cooled chamber. The crucible used would usually break because of the heat shock, but this happened after solidification of the metal.

b. Trial Melts: Alundum, magnesia, and graphite crucibles were not satisfactory. These crucibles were badly eroded by the metal and the castings produced were drossy and very brittle. A zirconia crucible showed less erosion, but the casting was not satisfactorily clean. Thoria crucibles were very slightly affected by the alloy and the castings produced in them were clean and sound.

Results obtained on four trial castings made with normal uranium and melted in a vacuum are shown in Table 3.

TABLE 3

Results from Trial Castings of U-Zr Alloys from Thoria Crucibles

Heat No.	Composition of Charge		% U by Analysis	Rockwell C Hardness	Machinability
	Zr (%)	U (%)			
U ⁿ Zr 15A	2.4	97.6	96.3	16-22	Good
U ⁿ Zr 15B	11.6	88.4	88.6	18-28	Good
U ⁿ Zr 15C	11.0	89.0	88.5	15-20	Good
U ⁿ Zr 14	20.2	79.8	77.4	16-22	Good

The thoria crucibles available produced castings approximately 1 in. in diameter x 1/4 in. thick. Castings 15A, 15B and 14 were cut into 1/4 in. x 1/4 strips using an 0.018 in. milling saw. These were then turned in a lathe to 0.210 in. diameter.

c. Trial Swaging at 300°C: The 0.210 in. pins were heated in oil to 300°C and swaged. The amount of reduction before the first cracks appeared is shown below.

No.	% Zr	Diameter of Last Pass	% Reduction
15A	2.4	0.109	73
15B	11.6	0.170	27
14	20.2	0.199	10

Considerable difficulty was experienced in handling the short pieces through the swaging machine, and an excessive quantity of material went into machining residue in the form of mill cuttings and turnings.

d. Trial Rolling at 600°C: Casting number Uⁿ Zr 15C was jacketed in a 1/2 in. thick steel plate. This was then heated to 600°C in lead and rolled to 1/8 in. thickness. The rolled sandwich was radiographed and the jacket was removed by end milling around the encased piece of alloy. Rolling data on this specimen are shown below.

No.	Original Thickness	Rolled Thickness	% Reduction	Condition of Rolled Plate
U ⁿ Zr 15C	0.254 in.	0.081 in.	68	Good

This method appeared to be the more practical one for the reduction of these small samples.

II. Fabrication of the Specified Enriched Alloy Specimens

The required series of alloys was accordingly made as small castings in thoria crucibles. Zirconium contents of these are given in Table 4, as computed from charge weight data and also as analyzed. The castings were cropped to provide material for analysis, as well as for the required as-cast pins. The main portions of the castings were jacketed in steel and rolled at 600°C, then divided and given the required heat treatments before machining of specimens. These are listed in Table 5. Table 6 shows the groupings of specimen materials in the individual pin-cushion mounts, together with the specimen code designations for zirconium content and metallurgical history. A final stress relief treatment at 575°C was given all specimens after machining had been completed.

TABLE 4

Chemical Composition of U-Zr Alloys for Pin Specimens

Casting No.	Zirconium Content			
	Nominal w/o	w/o Computed from Charge	Chemical Analysis	
			w/o	a/o
2-354	0(1)	-	- (2)	-
2-373	2	2.09	1.98	4.95
2-374	4	4.18	3.97	9.63
2-375	6	6.13	5.64	13.36
2-376	10	10.08	9.89	22.06
2-377	20	20.63	22.33	42.58

(1) Assay of uranium by mass spectrograph was $93.20 \pm 0.06\%$ U^{235} , $1.01 \pm 0.02\%$ U^{234} , balance U^{238} .

(2) Standard Deviation, $\pm 0.15\%$.

TABLE 5

Metallurgical Condition of Specimens

Condition No.	Mechanical Work	Heat Treatment Before Finish Machining ⁽¹⁾
1	As Cast	None
1a	Cast	800°C 1 hr; quench into 550°C bath 1 hr; "air" cool.
2	70% reduction in thickness ⁽²⁾	725°C 10 min; water quench, 575°C 2 hr; water quench.
2a	70% reduction in thickness ⁽²⁾	800°C 1 hr; quench into 550°C bath 1 hr; "air" cool.
3	70% reduction in thickness ⁽²⁾	725°C 1 hr; "air" cool.
3b	70% reduction in thickness ⁽²⁾	800°C 1 hr; cool in vacuum furnace.
4	70% reduction in thickness ⁽²⁾	575°C 1 hr; slow cool.

(1) All specimens were stress relieved at 575°C for 24 hours after finish machining.

(2) Rolled as "sandwich" plates at 600°C.

TABLE 6

Identification of Pin Materials With the Pin-Cushion Mounts
in Which the Specimens Were Grouped

Metallurgical Condition Nominal Zr Content (w/o)			Cast		Wrought (70% Reduction in Thickness at 600°C)				
			(1)	(1a)	(2)	(2a)	(3)	(3b)	(4)
			As-Cast	800-550 Quench	725 Quench	800-550 Quench	725 Anneal	800 Anneal	575 Anneal
(A)	0	Pins having these composi- tions were mounted in "pin-cushions" identified by numbers and letters appear- ing in the col- umns at right.			1, 2, G		2		2
(B)	2		5, G	H	1, 6, G	H		H	
(C)	4		5, I		1, 6, 7	I	7	I	7
(D)	6		4, 5, J		3, 6	J		J	
(E)	10		4		3				
(F)	20		4,8		3		8		8

APPENDIX II

Determination of Burnup

All specimens were irradiated in the CP-3' heavy water research reactor which operated, under normal conditions, at 275 kw and a water temperature of 35°C. Reported maximum thermal neutron flux to which the specimens were subject was 3.22×10^{12} nv.

Table 7 presents chronologically the available data on the various pin-cushion irradiations. Estimates of U^{235} depletion during each exposure are listed, no allowance being made for flux depression or self-shielding. These numbers, therefore, are greater than the actual burnups; however, they provide a relative scheme which can be normalized to correspond with actual burnup analyses.

TABLE 7

Irradiation Data and Computed Depletions of U^{235}

Irradiation Date		"Cushion" Numbers	CP-3' Request No.	Integrated Reactor Power (kwh)	Equivalent Irradiation Time at 275 kw (10^6 sec)	Estimated % Depletion of U^{235} (1)
In	Out					
5-22-51	6-18-51	1, 2	5136	127,828	1.67	0.27
7-17-51	8-20-51	1, 2	5167	162,244	2.12	0.35
9-24-51	11-11-51	3, 4	5041	215,809	2.83	0.45
11-28-51	3- 9-52	1, 5	5001	442,483	5.79	0.93
11-28-51	3- 9-52	7, 8	5002	438,410	5.74	0.59(2)
3-18-52	10-23-52	4, 6	5075	827,269	10.83	1.73
7-16-52	10-27-52	8	5214	385,501	5.05	0.52(2)
10- 7-52	10-27-52	1, 5(3)	5262	71,133	1.06	0.17
11-10-52	3- 2-53	4, 6	5282	409,035	5.35	0.86
11-24-52	4- 6-53	1, 5	5283	493,723	6.33	1.12
11-24-52	6-22-53	8	5286	793,515	10.39	0.68(4)
8- 3-53	1-18-54	G, H, I, J	5394	629,762	8.24	1.32
10- 5-53	1- 8-54	5, 6	5418	355,936	4.66	0.75

(1) Based on fission cross section of 500 barns and a reported flux of 3.22×10^{12} nv, except as noted.

(2) Reported flux, 2.04×10^{12} nv.

(3) No measurements taken; not counted as a separate exposure

(4) Reported flux, 1.30×10^{12} nv.

The estimates themselves cannot be considered very accurate. Even though an effort was made to have the specimens located in positions of maximum flux the buckling factors for actual vertical positions of the specimens in the reactor thimbles are not accurately known. The specimens were moved during the course of the irradiations, and adjacent experiments were repeatedly changed.

To provide a basis for assigning burnup numbers to the various exposures, direct determinations of total uranium burnup were made by Cs^{137} fission product analysis on fourteen of the specimens.⁽⁷⁾ These results are listed in Table 8, together with correlated U^{235} depletion estimates. It should be noted that the latter numbers are summations of the pertinent exposures listed in Table 7. They are plotted in Fig. 9 against the Cs^{137} results. Two of the points are of questionable value. Point 6D-2 is an erratic, quite likely due to the depletion estimate; point 3 was obtained from fragments of spallation found in cushion 3 and should be expected to deviate because of altered self-shielding. Four of the points, all from cushions I and J, agree together well but deviate somewhat from the average behavior of specimens irradiated in dry capsules. This may be explained by the more compact arrangement of pins in the wet capsule and the corresponding increase in flux depression. The remaining eight points support a fairly good straight line calibration which makes the combined flux depression and self shielding approximately 70%. Relative accuracy of the calibration is probably not better than 10%.

TABLE 8

Cesium-137 Analyses of Uranium Burnup in Selected Pins

Cushion Number ⁽¹⁾	Pin Number	Nominal Zirconium Content (w/o)	Exposures Accumulated Before Sampling	Estimated Per Cent Surface Depletion of U^{235}	Analyzed Average Per Cent Burnup of Total Uranium
1	A	0	4	2.84	0.86
2	B	0	2	0.62	0.148
3	Average	6-20	1	0.45	0.40
4	D-1	6	3	3.04	1.00
5	D-1	6	2	2.22	0.73
5	C-1	4	3	2.97	1.01
6	D-2	6	2	2.59	0.46
6	B-2	2	3	3.34	1.01
7	C-2	4	1	0.59	0.22
8	F-1	20	3	1.79	0.72
I	2	4	1	1.32	0.27
I	3	4	1	1.32	0.27
J	2	6	1	1.32	0.29
J	3	6	1	1.32	0.29

⁽¹⁾Cushions 1 to 8 were irradiated in dry capsules; G, H, I and J were in a wet capsule. Cushions 1 to 6 had a diameter of $1\frac{1}{4}$ in.; cushions 7 and 8 were $\frac{7}{8}$ in. in diameter.

Because of uncertainties in the computed uranium depletions, the eight Cs^{137} analyses mentioned above, together with an average of the four I and J determinations, are accepted at face value and used to assign average per cent uranium burnup numbers to the various specimens examined. The 6D-2 Cs analysis is also accepted; only 3 is ignored, and the uranium burnup numbers used for it and its related specimens are taken from the curve in Fig. 9. These selections have been listed in Table 9, and they are there converted into total atom burnup numbers which are proportional to the depletion numbers initially computed.

TABLE 9

Total Atom Burnup, Computed for the Various Cumulative Exposures of Pin Specimens

Cushion Number	Exposures Accumulated	Computed Depletion of U^{235} in Unperturbed Flux (%)	Assigned Average Total Uranium Burnup in Pins (%)	Average Per Cent Total Atom Burnup Accumulated Allowing for Zirconium Content					
				0 w/o Zr Factor 1.00	1.98 w/o Zr Factor 0.95	3.97 w/o Zr Factor 0.90	5.64 w/o Zr Factor 0.87	9.89 w/o Zr Factor 0.78	22.33 w/o Zr Factor 0.57
1	1	0.27	0.08	0.08	0.08	0.07			
	2	0.62	0.19	0.19	0.18	0.17			
	3	1.55	0.47	0.47	0.45	0.42			
	4	2.84	0.86	0.86	-	0.78			
2	1	0.27	0.07	0.07					
	2	0.62	0.15	0.15					
3	1	0.45	0.17				0.15	0.13	0.10
4	1	0.45	0.15				0.13	0.12	0.08
	2	2.18	0.72				0.62	0.56	0.41
	3	3.04	1.00				0.87	0.78	0.57
5	1	0.93	0.32		0.29	0.28	0.27		
	2	2.22	0.73		0.69	0.66	0.63		
	3	2.97	1.01		0.96	0.91	-		
6	1	1.73	-		-	-	-		
	2	2.59	0.46		0.44	0.41	0.40		
	3	3.34	1.01		0.96	0.91	-		
7	1	0.59	0.22			0.20			
8	1	0.59	0.24						0.14
	2	1.11	0.45						0.26
	3	1.79	0.72						0.41
G	1	1.32	0.28	0.28	0.27				
H	1	1.32	0.28		0.27				
I	1	1.32	0.28			0.25			
J	1	1.32	0.28				0.24		

APPENDIX III

Post-Irradiation Data

In Table 10 are listed the apparent per cent changes in length suffered by the pin specimens during the various reactor exposures. These were obtained by placing the pin-cushion mount on a face plate before and after irradiation and comparing the heights of pins with a dial gage. These apparent heights have been corrected for unaccountable differences in gage calibration by assuming in all cases that the zirconium reference pin at center position has not changed in length or in placement in the mounting socket.

Figures 10 to 23 are included in this appendix to record the appearance of all pin-cushions after the last reactor exposures to which they were subjected. In some cases two photographic records are presented, at differing exposure levels, because certain specimens were broken off at intermediate exposure levels to obtain fission product analyses of burnup. Captions of the figures usually refer to total uranium burnup instead of total atom burnup. This is because pins grouped in many of the cushions have differing alloy contents and have therefore not integrated the same total atom burnups. It is assumed that all pins in a cushion integrate the same total uranium burnup; these numbers are listed in Table 9 (Appendix II) and may be converted to total atom burnup data by referring to the columns which show the computed deviations caused by zirconium content.

TABLE 10

Changes in Measurements of Pin Lengths After Irradiations

Cushion Number	Pin Number and Heat Treatment	Nominal w/o Zr	Per Cent Growth of Pins							
			Exposure No. 1		Exposure No. 2		Exposure No. 3		Exposure No. 4	
			Measured	Corrected	Measured	Corrected	Measured	Corrected	Measured	Corrected
1	A(A-2)	0	5.2	5.1	9.7	9.4	20.8	14.9	19.6	19.6
	B(B-2)	2	4.5	4.4	7.5	7.2	13.3	7.4	-(1)	-
	C(C-2)	4	4.5	4.4	5.0	4.7	11.7	5.8	10.8	10.8
	Center	100	0.1		0.3	-	5.9		0	
2	A(A-4)	0	15.6	15.5	-(2)	-				
	B(A-2)	0	3.6	3.5	4.4	4.3				
	C(A-3)	0	9.8	9.7	-(2)	-				
	Center	100	0.1	-	0.1					
3	D-2	6	17.4	16.9						
	E-2	10	25.5	25.0						
	F-2	20	2.1	1.6						
	Center	100	0.5	-						
4	D-1	6	5.6	4.8	9.4	9.3	9.2	9.2		
	E-1	10	3.6	2.8	6.8	6.7	5.9	5.9		
	F-1	20	2.0	1.2	2.4	2.3	2.0	2.0		
	Center	100	0.8	-	0.1		0			
5	B-1	2	1.6	1.6	-4.8	-4.8	-2.5	-2.5		
	C-1	4	13.9	13.9	13.5	13.5	15.8	15.8		
	D-1	6	5.9	5.9	8.7	8.7	-(1)	-		
	Center	100	0	-	0		0			
6	B-2	2	3.3	3.3	8.8	8.8	10.1	10.1		
	C-2	4	2.0	2.0	4.2	4.2	6.1	6.1		
	D-2	6	11.4	11.4	8.1	8.1	-(1)	-		
	Center	100	0	-	0		0			
7	C-2	4	13.9	13.9						
	C-3	4	9.1	9.1						
	C-4	4	-(2)	-						
	Center	100	0	-						
8	F-1	20	6.4	6.4	7.0	6.9	7.4	7.4		
	F-3	20	3.2	3.2	0.4	0.3	4.2	4.2		
	F-4	20	5.9	5.9	5.4	5.3	5.8	5.8		
	Center	100	0	-	0.1		0			
G	1(A-2)	0	4.8							
	2(B-1)	2	-0.6							
	3(B-2a)	2	4.5							
H	1(B-1a)	2	-4.6							
	2(B-2a)	2	7.2							
	3(B-3b)	2	5.9							
I	1(C-1)	4	8.8							
	2(C-2a)	4	-4.3							
	3(C-3b)	4	-2.9							
J	1(D-1)	6	7.2							
	2(D-2a)	6	-2.7							
	3(D-3b)	6	-1.6							

(1) Pin previously removed for burnup analysis.

(2) Pin disintegrated during irradiation.

Figure 10. Appearance of Cushion 1 after Irradiation. All pins had 300°C roll, 725°C quench, 575°C anneal.

- (a) After 0.46% total U burnup.
 (b) Fracture surface of pin broken at 0.46% total U burnup.
 (c) After 0.86% total U burnup.

Figure 11. Appearance of Cushion 2 after Irradiation. Pins are 100% U rolled at 300°C. Heat Treatments are: α a-575°C anneal; β q-725°C quench, 575°C anneal; β a-725°C anneal.

(a) After 0.07% total U burnup.

(b) After 0.15% total U burnup.

Figure 12. Appearance of Pins in Cushion 3 after Irradiation to 0.17% Total U Burnup. All pins had 300°C roll, 725°C quench, 575°C anneal.

(a)

U-6% Zr

12209

(b)

U-10% Zr

12210

(c)

U-20% Zr

12211

Figure 13. Appearance of Cushion 4 after Irradiation to 1.00% Total U Burnup. All specimens as-cast.

(a)

U-10% Zr

Zr

U-20% Zr

14467

U-6% Zr

5-X

(b)

U-10% Zr

(c)

U-20% Zr

17980, 17981

34-X

Figure 14. Appearance of Cushion 5 after Irradiation to 0.73% (a) and 1.01% (b,c) Total U Burnup. All pins as-cast.

(a)

U-4% Zr

14468

U-2% Zr

Zr

U-6% Zr

5-X

(b)

U-2% Zr

(c)

U-4% Zr

17983, 17984

34-X

Figure 15. Appearance of Cushion 6 after Irradiation to 0.78% (a) and 1.01% (b,c) Total U Burnup. All pins had 300°C Roll, 725°C quench, 575°C anneal.

(a)

U-4% Zr

14469

U-6% Zr

Zr

U-2% Zr

5-X

(b)

U-2% Zr

(c)

U-4% Zr

17986, 17987

34-X

Figure 16. Appearance of Cushion 7 after Irradiation to 0.22% Total U Burnup (0.20% Total atom burnup). All specimens U-4% Zr, rolled at 300°C and heat treated.

(a)

 α a

Zr

 β q

12557

β a

5-X

(b)

 β a

(c)

 α a

17989, 17991

34-X

Figure 17. Appearance of Cushion 8 after Irradiation to 0.72% Total U Burnup (0.41% Total atom burnup). All specimens U-20% Zr, condition as indicated.

(a)

 β α

Zr

 α a

14470

5-X

As

Cast

(b)

 β a

17992, 17993

34-X

(c)

 α a

Note that the beta annealed specimen has lost a longitudinal chip.

Figure 18. Appearance of Cushions G and H after Irradiation to 0.28% Total U Burnup.

a

b

c

(See

Fig.

19)

d

e

f

(See

Fig.

20)

18003, 18007

4-1/4-X

Upper specimen (a) is 100% U, 0.28% total atom burnup; all others are U-2% Zr, 0.27% total atom burnup. All specimens except (b) and (f) rolled at 300°C before heat treatment; (b) and (f) cast. Heat treatments are as follows (Table 5):

(a) and (c) 2; 725°C quench, 575°C anneal (b) 1; as-cast. (d) 3b; 800°C slow cool. (e) and (f) 2a; 800°-550°C interrupted quench.

- (a) 100% U, 725°C quench, 575°C anneal; 0.28% Total atom burnup.
- (b) U-2% Zr, as-cast; 0.27% Total atom burnup.
- (c) U-2% Zr, 725°C quench, 575°C anneal; 0.27% Total atom burnup.

38 Figure 20. Free Ends of Pins from Cushion H after 0.28% Total U Burnup.
(See Fig. 18, d, e, f.) (0.27% Total Atom Burnup.)

d

e

f

18010, 18009, 18008

34-X

- (d) U-2% Zr, 800°C slow cool.
- (e) U-2% Zr, 800°-550°C interrupted quench.
- (f) U-2% Zr (cast), 800°-550°C interrupted quench.

Figure 21. Appearance of Cushions I and J after Irradiation to 0.28% Total U Burnup.

b

c

(See
Fig.
22)

d

e

f

(See
Fig.
23)

18011, 18015

4-1/4-X

Specimens (a) (b) (c) are U-4% Zr, 0.25% total atom burnup; (d) (e) (f) are U-6% Zr, 0.24% total atom burnup. All specimens except (a) and (d) rolled at 300°C before heat treatment; (a) and (d) as-cast. Heat treatments are as follows (Table 5):

- (a) and (d) 1; as-cast.
- (b) and (e) 2a; 800°-550°C interrupted quench.
- (c) and (f) 3b; 800°C slow cool.

Figure 22. Free Ends of Pins from Cushion I after 0.28 Total U Burnup (0.25% Total Atom Burnup). (See Fig. 21, a, b, c.)

a

b

c

18012, 18013, 18014

34-X

- (a) U-4% Zr, as-cast.
- (b) U-4% Zr, 800°-550°C interrupted quench.
- (c) U-4% Zr, 800°C slow cool.

Figure 23. Free Ends of Pins from Cushion J after 0.28% Total U Burnup (0.24% total atom burnup). (See Fig. 21, d, e, f).

d

e

f

18016, 18017, 18018

34-X

- (d) U-6% Zr, as-cast.
- (e) U-6% Zr, 800°-550°C interrupted quench.
- (f) U-6% Zr, 800°C slow cool.