UNCLASSIFIED

AECD-3794

Subject Category: METALLURGY AND CERAMICS

0

UNITED STATES ATOMIC ENERGY COMMISSION

COMPARATIVE BEHAVIOR OF A 1.8 ATOM %
CHROMIUM-URANIUM ALLOY AND NORMAL
URANIUM ON THERMAL CYCLING

By
D. W. White

July 14, 1950

Knolls Atomic Power Laboratory
Schenectady, New York

Technical Information Service, Oak Ridge, Tennessee

UNCLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

1 -

Date Declassified: December 6, 1955.

This report was prepared as a scientific account of Government-sponsored work and is made available without review or examination by the Government. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights. The Commission assumes no liability with respect to the use of, or for damages resulting with respect to the use of any information, apparatus, method, or process disclosed in this report.

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

Issuance of this document does not constitute authority for declassification of classified material of the same or similar content and title by the same authors.

Printed in USA, Price 20 cents. Available from the Office of Technical Services, Department of Commerce, Washington 25, D. C.

2

GENERAL ELECTRIC COMPANY

Knolls Atomic Power Laboratory
Schenectady, New York

Comparative Behavior of a 1.8 atom % Chromium-Uranium Alloy and Normal Uranium
on Thermal Cycling

July 14, 1950

by

D. W. Whit-

Contract No. W-31-109 Eng. 52

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

Comparative Behavior of a 1.8 atom % Chromium-Uranium Alloy and Normal Uranium on Thermal Cycling

Introduction:

Chromium as an alloying element in uranium has the effect of retarding the rate of the beta-to-alpha transformation. This characteristic allows one to transform uranium containing a small amount of chromium isothermally and thus achieve a considerable degree of grain size control that is not possible in unalloyed uranium by any known heat treatment.

Although the advantages of a suitably heat-treated U-Cr alloy are recognized from the standpoint of grain size, nothing is known about the dimensional stability of this material. It was the object of the test described herein to evaluate the dimensional stability under thermal cycling conditions of a 1.8 at. % Cr-U alloy (0.4 wt. % Cr) having various isothermal heat-treatments and to compare it with that of pure uranium in three different conditions. This particular alloy composition was chosen as it is the alloy whose rates of isothermal transformation have been studied most extensively at KAPL.

Abstract:

A thermal cycling test, consisting of 500 cycles between 100° and 500°C, was conducted on (a) six pairs of 1.8 at. % Cr-U alloy specimens that had been heat treated by isothermal transformation at temperatures varying from 449° to 614°C, (b) an as-alpha-swaged uranium specimen, (c) a uranium specimen that had been quenched from beta, and (d) a uranium specimen that had been quenched from beta and then annealed in high alpha.

The chromium alloy was dimensionally stable to 0.1% or less. The heat treated uranium specimens expanded by about 1%, while the as-worked uranium elongated nearly 19%.

The chromium alloy retained relatively smooth surfaces while the uranium specimens roughened as a result of thermal cycling. The slight amount of surface roughening that did occur in the U-Cr specimen group was more prevalent in those specimens that had been transformed at the higher temperatures.

Metallographically, the chromium alloy was characterized by more uniform grain size and structure than the heat-treated uranium. Increasing grain

refinement was achieved by lowering the temperature of isothermal transformation. Transforming the chrome alloy at 581°C produced a grain size no larger than the maximum grain size of heat treated uranium.

Hardness and density measurements were made; data are tabulated.

Preparation of Test Samples:

The starting materials were (1) a 3/4" dia casting of U-Cr alloy heat 2A-15, whose chromium content was nominally 2.0 at. %, but actually 1.8 at. % by chemical analysis, and (2) a Hanford aluminum-canned slug (triple-dipped) which was de-canned and machined to 3/4" dia. After an anneal of several hours at 550°-600°C, the two pieces were swaged to 0.434" dia., with intermediate anneals of 1 hour at 550°C after every 50% reduction. The swaging temperature was 275°C.

The swaged bars were cut into 17/32" lengths, four of which were from the uranium bar and twelve from the U-Cr alloy bar. One of the uranium pieces was set aside for a pilot sample, as will be described later. The twelve alloy samples were "normalized" by heating in vacuum to 725°C and furnace cooling, the purpose of which was to saturate the beta phase with chromium.

The U-Cr samples were heat treated in duplicate, using six different temperatures of isothermal transformation. The lengths of time the samples were held at these temperatures were more than sufficient to allow complete transformation, based on data obtained in a previous study of the beta decomposition rates of this alloy (see Fig.1). Two of the pure uranium samples were beta quenched and one of these was subsequently given a high alpha anneal; the third uranium sample was left in the as-swaged condition. All the heat treatments were performed in molten tin-lead eutectic baths. A thin application of silicone grease to the surface of the specimens prevented attack by the heat treating baths. The heat treatments of the fifteen specimens are listed in detail in Table I.

After heat treatment, the specimens (plus the pilot sample) were machined to 0.400" dia. by 0.500" long. In addition, a hole was drilled half way through the pilot sample to serve as a thermocouple well.

Thermal Cycling Test:

The specimens were enclosed in duplicate and at random in eight sealed Pyrex tubes, 5/8" O.D. x 2 1/2" long, containing a partial pressure of helium. A chromel-alumel thermocouple was inserted into the pilot sample and was brought out through the Pyrex envelope by means of tungsten-to-glass seal.

The thermal cycling was performed in an apparatus designed and built by Mr. H. Hirsch of KAPL. A cylindrical metal basket, in which the eight Pyrex tubes were stacked around the periphery, is automatically lowered and raised

in and out of a furnace. In the "out" position cooling is facilitated by two fans. The cycles are temperature controlled and operate between pre-set temperature limits. The fifteen specimens of concern here were cycled between 100°C and 500°C, as represented by the temperature of the pilot sample. The furnace was set at a higher temperature than 500° - actually about 600° - to speed up the heating half of the cycle. The duration of the test was 500 cycles, consuming about 5 days. The average length of a cycle was thus about 15 minutes, of which the heating portion was slightly longer than the cooling portion.

Results and Discussion:

The results are listed in Table II. The dimensional stability is indicated by the percentage change in length and in diameter during cycling. The dimensional changes of the uranium-chromium alloy specimens, except #6 and #12, are seen to be in the order of 0.1% and in many cases much less. The accuracy of measurement is no greater than this amount, as the actual changes amounted to only a few ten-thousandths of an inch, and therefore the dimensional changes may be insignificant.

Specimens #6 and #12 exhibited a small, but significant, shrinkage in length and in diameter, suggesting the presence of retained beta before cycling and the subsequent transformation to alpha during cycling. These specimens had been isothermally heat treated well above the "nose" of the time-temperature transformation curve (Figure 1) in a region where the rate of transformation is very temperature-sensitive. Although they were held at temperature for a length of time that appeared to be sufficient to produce complete transformation, it is highly possible that the actual temperature was a few degrees higher than indicated and hence a considerably longer time was required to transform the material. That these specimens contained beta phase at the start of the cycling test is indicated by the following observations:

1. The grain size was much smaller at the end of cycling than at the beginning (photomicrographs #257 and #485 in Figure 2), indicating that retained beta transformed to alpha during the cycling test at temperatures where a smaller-grained product is obtained.
2. The two specimens exhibited surface checks and cracks, very likely resulting from internal stresses set up by non-uniform rates of transformation over the cycling temperature range. Cracking is typical of thermally cycled specimens having had retained beta at the start.
3. The dimensional and volume shrinkage as mentioned above. Alpha is more dense than beta.
4. The hardness of specimens #6 and #12 was significantly lower than that of the other U-Cr specimens before cycling. After cycling, it was about the same as that of the other specimens.

While the isothermally transformed U-Cr specimens proved to be very stable dimensionally, the heat treated uranium specimens (#13 and #14) expanded by about 1%, or an order of magnitude greater than the U-Cr alloy. The as-alpha-swaged uranium specimen (#15) lengthened nearly 19% and decreased in diameter by as much as 7%.

The values for dimensional change of specimens #13, #14, and #15 listed in Table II are somewhat fictitious, as they give apparent volume increases of as much as 4.17%, 4.09% and 5.36%, respectively, for the three specimens. Since the densities of these specimens after cycling were normal for uranium, the volume increases are obviously not real and arise from the fact that the micrometer measurements of the cycled specimens were somewhat high due to bumpy surfaces and cupped ends. The calculated volume changes of the U-Cr specimens are negligible (except for specimens #6 and #12, as previously discussed) because those specimens had relatively smooth surfaces.

In addition to dimensional stability, the chrome alloy specimens proved to have greater surface stability in thermal cycling than the pure uranium. While the uranium specimens acquired rough, bumpy surfaces, the U-Cr remained relatively unaffected (except specimens #6 and #12, as previously discussed). This behavior is related to the grain structure. The U-Cr specimens possessed essentially uniform grain sizes and structures, due to the fact that they were transformed isothermally, while the heat treated uranium specimens had a wide range of grain sizes and shapes. Specimen #13 had heavily-twinned grains with jagged boundaries, as is typical of beta-quenched uranium; after cycling, the twin markings were less prevalent but the grain size variation was still present. Specimen #15 displayed a heavily cold-worked structure, which was converted by the cycling test into a fined-grained structure. The great length increase and the ribbed surface in specimen #15 resulted from the highly preferred orientation that exists in uranium that has been cold worked by swaging.

Within the U-Cr specimen group itself, there was a tendency for slightly increased surface roughening with an increase in temperature at which a specimen had been isothermally transformed. Again, this behavior is related to grain size. Because of a higher rate of nucleation and a lower rate of grain growth at the lower temperatures, grain size decreases with decreasing temperature at which a specimen is transformed.

The discovery that all the chromium alloy specimens (except #6 and #12) were equally stable dimensionally and did not vary greatly in surface stability has an important, practical implication. It means that fairly large, massive sections of a U-Cr alloy may be heat treated. Despite the fact that, in quenching a large piece to the temperature at which it is to be isothermally transformed, part of the interior may not cool sufficiently rapidly to escape the "nose" of the C-curve (Figure 1), the small amount of product that transforms at the "nose" temperature (about 575°C) possesses good dimensional and surface stability. The only danger lies in the susceptibility to cracking during heat treatment if a substantial percentage of the material transforms at temperatures above the desired isothermal transformation temperature. The maximum size of a

piece that can be heat treated without cracking cannot be predicted at this stage. It is speculated that, if a piece is very large and massive, it should be isothermally transformed at a temperature not far below the "nose" of the C-curve, in order to reduce the temperature range over which the piece will actually transform from center to outside.

Densities were measured on all the specimens after cycling. They are listed in Table II and appear to be normal. Unfortunately, there are no density values before cycling for comparison.

Hardness values before and after cycling are listed in Table II. It is noted that the chromium alloy was found to be harder than the pure uranium. Within the U-Cr alloy group, there tended to be a slight increase in hardness with decreasing grain size-i.e., with decreasing temperature of isothermal transformation.

Conclusions:

1. Isothermally heat treated 1.8 at.% Cr-U alloy has greater dimensional and surface stability under thermal cycling (100°-500°C, 500 cycles) than as-worked or heat-treated uranium.

2. By isothermal heat treatment, considerably finer and more uniform grain size can be produced in a 1.8 at.% Cr-U alloy than can be achieved in normal uranium by heat treatment. Grain size decreases with decreasing temperature at which transformation is allowed to take place.

3. The recommended heat treatment for a 1.8 at.% chromium-uranium alloy is to heat well into the beta - say, 700°-720°C - and hold there for a length of time greater than is necessary to secure temperature equilibrium. Then, quench rapidly to some lower temperature and hold at that temperature for a sufficient length of time to assure complete transformation to alpha. Subsequent cooling may be done in any convenient fashion.

Recommended temperatures to which a U-Cr piece should be quenched and the corresponding lengths of time for which they should be held isothermally, unless the piece is extremely large and massive, are listed in order of preference, as follows: 450°C for 3 hours, 475°C for 2 hours, 500°C for 1 hour, 525°C for ½ hour, 550°C for 15 minutes, 575°C for 10 minutes. The lower temperatures in the above list may not be satisfactory for large pieces.

9

TABLE I

Sample	Material	Heat Treatment							
#1 and #7	U-Cr	15mins at 714°C. Quenched to 449°C. and held 285 mins W.Q.							
#2 and #11	U-Cr	19 "	"	713°	"	"	489°	"	"
#3 and #8	U-Cr	23 "	"	715°	"	"	513°	"	"
#4 and #9	U-Cr	15 "	"	711°	"	"	547°	"	"
#5 and #10	U-Cr	15 "	"	723°	"	"	581°	"	"
#6 and #12	U-Cr	15 "	"	719°	"	"	614°	"	"
#15	U	as-alpha-swaged (No heat treatment)							
#13	U	10 minutes at 734°C. Water quenched							
#14	U	same as #13, plus 45 minutes at 579°/582°C.							

TABLE II

Results of Thermal Cycling 500 Times Between 100° and 500°C

Specimen Number	Material	Treatment	Dimensional change - %		Surface appearance after cycling	Density after cycling	Rockwell C Hardness	
			Diam.	Length			Before cycling	After cycling
1	U-Cr	Isoth transformed at 449°C	+0.125	+0.060	Smooth	18.84	34.3	24.3
7	U-Cr		+0.125	+0.100	Slightly pitted	18.82	33.1	32.7
2	U-Cr	Isoth transformed at 489°C	+0.100	+0.020	Slightly pitted	18.80	33.0	26.5
11	U-Cr		+0.075	-0.080	Very smooth	18.81	33.0	27.8
3	U-Cr	Isoth transformed at 513°C	+0.050	0	Very smooth	18.80	32.6	27.5
8	U-Cr		+0.100	0	Smooth	18.82	32.1	26.3
4	U-Cr	Isoth transformed at 547°C	+0.025	+0.020	Pitted	18.82	27.3	27.5
9	U-Cr		+0.050	+0.020	Slightly pitted	18.83	27.6	25.8
5	U-Cr	Isoth transformed at 581°C	+0.125	-0.020	Pitted; some checks	18.82	27.7	24.1
10	U-Cr		-0.075	+0.020	Pitted	18.82	31.6	25.5
6	U-Cr	Isoth transformed at 614°C	-0.275	-0.340	Profuse checking	18.81	21.0	24.5
12	U-Cr		-0.350	-0.399	Profuse checking	18.82	21.8	25.8
15	U	As- α -swaged	{5.776 -7.025}	{+15.375 +18.670}	Woody, ribbed surface	18.84	24.0	7.6
13	U	β -quenched	{1.449 +1.699}	{+0.460 +0.720}	Warty surface; some pitting	18.78	14.8	12.0
14	U	β -quenched & α annealed	{0.700 +1.499}	{+0.879 +1.039}	Rough surface; slight pitting	18.92	3.0	7.5

Note: The double values of dimensional change for specimens 13, 14 and 15 represent the maximum and minimum, as the specimens were not uniform in diameter nor square at the ends after cycling.

#234 Specimen #7
Isothermally Transformed at 449°C.
Before and After Cycling

#236 Specimen #11
Isothermally Transformed at 489°C.
Before and After Cycling

#482 Specimen #8
Isothermally Transformed at 513°C.
Before and After Cycling

#253 Specimen #4
Isothermally Transformed at 547°C.
Before and After Cycling

FIGURE 2
Cross-sectional Photomicrographs
of
1.8 At. % Cr-U Alloy Thermal Cycling Specimens

100X Magnification
Polarized Light

#254 Specimen #5
Isothermally Transformed at 581°C.
Before and After Cycling

#257 Specimen #12
Isothermally Transformed at 614°C.
Before Cycling

#485 Specimen #12
Isothermally Transformed at 614°C.
After Cycling

100X Magnification
Polarized Light

FIGURE 2 (cont.)
Cross-sectional Photomicrographs
of

1.8 At. % Cr-U Alloy Thermal Cycling Specimens

#238 - Before Cycling

#477 - After Cycling

Specimen #15 - As-Alpha-swaged

#237 - Before Cycling

#479 - After Cycling

Specimen #13 - Beta-quenched

#256 - Before Cycling

#478 - After Cycling

Specimen #14 - Beta-quenched + High-Alpha Annealed

100X Magnification
Polarized Light

FIGURE 3
Cross-sectional Photomicrographs of Uranium Thermal Cycling Specimens

**THIS PAGE
WAS INTENTIONALLY
LEFT BLANK**