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12-30-64

UCRL-7824

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**ISOTHERMAL TRANSFORMATION
STUDY OF SOME URANIUM-BASE ALLOYS**

Livermore, California

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UCRL-7824
Metals, Ceramics,
and Materials, UC-25
TID-4500 (34th Ed.)

UNIVERSITY OF CALIFORNIA
Lawrence Radiation Laboratory
Livermore, California

Contract No. W-7405-eng-48

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ABSTRACT

Isothermal time-temperature-transformation characteristics are presented for the gamma phase of alloys of uranium with molybdenum, niobium, and zirconium.

The changes in hardness and microstructure are described and correlated with the time, as determined by resistivity measurements, to initiate transformation at a given temperature.

INTRODUCTION

This summary of the time-temperature-transformation characteristics of the gamma phase covers uranium-base alloy compositions which were originally surveyed in a search for strong, fabricable, gamma-stable alloys. Included in this research were binary and ternary compositions of uranium with molybdenum, niobium, or zirconium.

The diagrams are exploratory in nature and indicate the times for initiation of the gamma-phase transformation. For the most part, the study was confined to the temperature region between 450°C and the eutectoid temperature because this was the area of most rapid decomposition of the gamma phases. When Zr was introduced to form ternary alloys, transformation proceeded more rapidly at the lower temperatures. Therefore, the majority of the experiments were conducted below 450°C with Zr-containing alloys. Also, because there was evidence of different modes of gamma-phase transformation, depending on temperature, the alloys containing Zr were studied in greater detail. The changes in hardness and structure of one alloy, the U-10 Nb-1 Zr, are given in this report because the data indicate the possibility of controlling the mechanical properties of

uranium alloys by suitable alloying and heat treatment. Another such example has been described in UCRL-7869. Alloys were prepared by consumable-arc-melting electrodes composed of the pure component metals. These first argon melts were remelted before working.

EXPERIMENTAL PROCEDURE

Materials

The nominal analyses of the alloys studied are given in Table I. The ingots were hot-forged and hot-rolled to plate and homogenized by gamma-annealing above 800°C.

Transformation Measurements

The technique for measurement of the change in resistivity during transformation of the gamma phase has been described elsewhere.¹ The data thus obtained are the bases for the various "C" curves and have been corroborated by hardness and metallographic measurements.

The metallographic samples were small cubes which had been gamma-annealed and quenched directly to the transformation temperature. Specimens were polished and etched; either cathodically in argon, or by cathodic etching followed by electrolytic etching in a phosphoric-acid-ethanol-ethylene-glycol solution. The latter solution was very useful in revealing details of pearlitic structure obtained at higher temperature levels. At lower temperatures it usually was not possible to follow the course of transformation by optical metallography.

X-ray diffraction and hardness measurements confirmed identification of phases and followed the course of transformation at the lower temperatures (below 450°C). The x-ray samples and hardness samples were prepared in the same manner as the metallographic samples and electropolished. Hardness measurements were made using a 500-gram load and diamond pyramid indenter.

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¹UCRL-7869 - The Properties of a Metastable Gamma-phase Uranium-Base Alloy (1964).

Table I. Nominal composition of alloys.

No.	Analysis (w/o)				U
	Mo	Nb	Zr	C	
1	3	-	-	a	Balance
2	5	-	-		"
3	7	-	-		"
4	8	-	-		"
5	10	-	-		"
6	12	-	-		"
7	-	5	-		"
8	-	8	-		"
9	-	10	-		"
10	5	5	-		"
11	5	-	1		"
12	5	-	3		"
13	7	-	1		"
14	7	-	2		"
15	7	-	3		"
16	10	-	1		"
17	10	-	2		"
18	10	-	4		"
19	10	-	6		"
20	-	9	1		"
21	-	10	1		"
22	-	10	4		"

^aCarbon < 90 ppm in all alloys.

Results and Discussion

Hardness measurements, x-ray diffraction patterns, and metallographic observations are consistent in the indication of transformation initiation. The resistometric method was probably the most sensitive, and for high-temperature determinations was in good agreement with metallographic results. At lower temperatures, metallography was inferior and the resistometric method and hardness measurements were most reliable. X-ray diffraction was used mostly for the purpose of phase identification.

Group I. U Binary Alloys - 3 to 12 w/o Mo

This series of "C" curves, Fig. 1, clearly reflects the influence of Mo content upon decomposition of the gamma phase. With less than 5 w/o Mo it would be difficult to retain gamma phase without rapid quenching. On the other hand, with 8 or 10 w/o Mo, a cooling rate of about 10°C per minute would allow retention of gamma phase at room temperature (see Fig. 2).

Metallographic examination shows that for these alloys, the transformation of the gamma phase above 450°C occurs by a nucleation and growth of cells or plates of alpha in the gamma matrix. Figures 3 and 4 illustrate the lamellar product. Apparently, after 6 hours at 500°C, the gamma phase has completely transformed.

Hardness changes as the transformation progresses are not too pronounced for heat treatments in this 500°C temperature range. Also, an average of the readings is necessary when transformed areas and nontransformed areas are encountered, because of the differences in hardness of such areas.

Group II. U Binary Alloys - 5, 8, and 10 w/o Nb

Three alloys of uranium with niobium show that the gamma-phase stability increases with increasing Nb content (Fig. 5). It is also apparent that Nb is not as effective as Mo in preventing decomposition of the gamma phase in the region between 450°C and the eutectoid temperature. It takes a 10 w/o Nb addition to achieve the same stability as is observed for 5 w/o Mo.

Fig. 1. Isothermal transformations of U-Mo alloys.

Transformation of gamma-phase 8 Mo-U alloy

Fig. 2. Gamma-quenched from 800°C, DPH-275 (400X).

Fig. 3. Gamma-quenched and transformed at 500°C for 4 hours, DPH-330 (400X).

Fig. 4. Gamma-quenched and transformed at 500°C for 6 hours, DPH-395 (400X).

Fig. 5. Isothermal transformations of U-Nb alloys.

The transformation of the gamma phase occurs by development of the lamellar $\alpha + \gamma$ phases. The final product is the Nb-rich solid solution of U in Nb (called γ') plus the α phase. Various stages in the transformation of the γ phase are seen in Figs. 6, 7, and 8.

Again, the hardness changes for these transformations are not exceptionally great, but are larger than found in the U-Mo alloys.

Group III. U-Nb-Mo Ternary Alloys

The gamma phase of one alloy examined, i. e., the 5 Nb-5 Mo composition, decomposes at a rate in between those for the binary 5 Nb and the binary 5 Mo as shown in Fig. 9.

Constitutional changes during transformation are similar to those occurring in each of the binary alloys of U with Nb or Mo. Figures 10, 11, and 12 illustrate the sequence in decomposition of the gamma-phase after one hour and five hours.

Group IV. U-Mo-Zr Ternary Alloys

When Zr is added to U-Mo-base alloys of 5, 7, and 10 w/o Mo content, the transformation kinetics observed in Figs. 13, 14, and 15, respectively, are obtained. The speeding up of the gamma-phase transformation by the addition of Zr to the binary U-Mo alloys is ascribed to the formation of ZrMo_2 compound. This reaction effectively removes the Mo necessary to render the gamma-phase metastable.

Figures 16 and 17 are micrographs of the as-quenched and transformed 10 Mo alloy containing 1 w/o zirconium. The ZrMo_2 is clearly visible in both photographs. The calculated tie-up of the Mo by Zr in this alloy is roughly 2 w/o, and the "C" curve is nearly the same as one for 7.5 w/o Mo alloy; so the agreement is fairly good.

Group V. U-Nb-Zr Ternary Alloys

Zirconium increases the time for initiation of gamma-phase decomposition of the U-Nb alloy if the isothermal treatment is above 450°C. Below 450°C the zirconium tends to accelerate the reaction. A typical case of this sort, the 7.5 Nb-2.5 Zr alloy, has been studied in detail and reported in another paper, UCRL-7869, 1964. Other examples of the complex transformation characteristics of these ternary alloys may be seen in Figs. 18, 19, and 20.

Transformation of gamma-phase 10 Nb-U alloy

Fig. 6. Gamma-quenched from 800°C, DPH-260 (400×).

Fig. 7. Gamma-quenched and transformed at 500°C for 0.5 hour, DPH-300 (400×).

Fig. 8. Gamma-quenched and transformed at 500°C for 2 hours, DPH-475 (400×).

Fig. 9. Isothermal transformations of U-5 Nb, U-5 Nb-5 Mo, and U-5 Mo alloys.

Transformation of gamma-phase 5 Mo-5 Nb-U alloy

Fig. 10. Gamma-quenched from 800°C, DPH-240 (400X).

Fig. 11. Gamma-quenched and transformed at 500°C for 1 hour, DPH-485 (400X).

Fig. 12. Gamma-quenched and transformed at 500°C for 5 hours, DPH-495 (400X).

Fig. 13. The effect on the isothermal transformation of Zr additions to U-5 Mo alloy.

Fig. 14. The effect on the isothermal transformation of Zr additions to U-7 Mo alloy.

Fig. 15. The effect on the isothermal transformation of Zr additions to U-10 Mo alloy.

Transformation of gamma-phase U-10 Mo- 1 Zr alloy

Fig. 16. Gamma-quenched from 800°C, DPH-315 (400X)

Fig. 17. Gamma-quenched and transformed at 500°C for 1.5 hours, DPH-485 (400X).

Fig. 18. Isothermal transformation of 9 Nb-1 Zr-U alloy.

Fig. 19. Isothermal transformation of 10 Nb-4 Zr-U alloy.

Fig. 20. Isothermal transformation of 10 Nb-1 Zr-U alloy.

These alloys are variations on the more gamma-stable base composition of U-10 Nb, and are quite similar in their characteristic behavior. The tendency to split the high-temperature "C" curves (above 450 to 475°C) into two "C" curves, which is very apparent in the 10 Nb-4 Zr alloy, is not explained by any of the present experimental evidence. This alloy is more gamma-stable in the high-temperature region than the two alloys with lower zirconium content; i. e., 9 Nb-1 Zr and 10 Nb-1 Zr.

From slightly above 400°C to approximately 475°C, a second minor "C" curve appears. Below 400°C the reaction time increases rapidly toward a nose around 300 to 350°C. The position of this nose moves toward shorter time intervals for either less niobium or higher zirconium content.

It is quite clear from metallographic studies that the transformation at the higher temperatures (450°C to the eutectoid) occurs by the now familiar nucleation and growth of plates in the γ phase (Figs. 21 through 29). The use of polarized light helps to delineate the new phase in certain cases.

In the region of the small middle "C" curve, metallographic structures take on an odd appearance; ranging from a vein-like or incipient cellular structure such as shown in Figs. 31, 34, and 35 to the twin-like plate structure of Figs. 32 and 36. In most of these cases polarized light was required to bring out any contrasting areas.

In the region of the nose of the lower "C" curve, structures like those of Figs. 37 and 38 appeared. To reveal these martensitic distorted areas, polarized light was again necessary.

Hardness measurements and x-ray diffraction were used to confirm the metallographic structures, or to reveal changes not visible under the microscope. These data are summarized in Table II. The changes in hardness with transformation of the gamma-phase are shown in Fig. 39. It is apparent from these curves that the transformation is very rapid between 350°C and 450°C and could very well be martensitic in nature. The hardening of the alloy is also greatest in this region. For the time of 8 hours employed in the 300°C heat treatments only nominal hardening occurred; no evidence of transformation could be seen in the microstructures at this temperature.

Table II. Effect of isothermal heat treatment of a gamma-quenched 10 Nb-1 Zr - U alloy.

No.	Heat Treatment	D. P. H.	Metallographic and diffraction report
23	As gamma-quenched from 900°C	260-270	1 phase - γ
19	10 min at 300°C	325	1 phase - γ
20	0.5 h at 300°C	333	1 phase - γ
21	1 h at 300°C	335	1 phase - γ
22	8 h at 300°C	350	1 phase - γ (distorted)
15	10 min at 350°C	266	1 phase - γ
16	0.5 h at 350°C	310	1 phase - γ
17	1 h at 350°C	367	2 phases - γ' + distorted α
18	8 h at 350°C	495	2 phases - γ' + α (martensitic-type twins)
24	0.5 h at 410°C	330	1 phase - γ
25	1 h at 410°C	375	2 phases - γ' + veined
26	8 h at 410°C	550	2 phases - γ' + α veined
12	0.5 h at 450°C	335	1 phase - γ
13	1 h at 450°C	375	1 phase - γ
14	8 h at 450°C	550	2 phases - γ' + martensitic-type twins (α)
8	10 min at 500°C	277	1 phase - γ
9	0.5 h at 500°C	335	2 phases - γ' + α (grain boundary pearlite)
10	1.0 h at 500°C	340	2 phases - γ' + α (20-25%)
11	8 h at 500°C	495	2 phases - γ' + α (75%)
4	10 min at 550°C	310	1 phase - γ
5	0.5 h at 550°C	295	2 phases - γ' + α (~ 10%)
6	1 h at 550°C	335	2 phases - γ'' + α (~ 25%)
7	8 h at 550°C	455	2 phases - γ' + α (~ 100%)
29	0.5 h at 575°C	260	2 phases - γ' + α (~ 5%)
30	8 h at 575°C	400	2 phases - γ' + α (~ 90%)
1	0.5 h at 600°C	297	1 phase - γ
2	1 h at 600°C	275	2 phases - γ' + α (starting to form)
3	8 h at 600°C	375	2 phases - γ' + α (~ 50%)

SUMMARY AND CONCLUSIONS

Niobium and molybdenum stabilize the gamma phase of uranium. The greater the addition of either element to form a binary alloy with uranium, the longer is the delay in the initiation of the gamma-phase transformation. During heat treatment at temperatures ranging from 450°C to the eutectoid temperature, the "nose" of the T-T-T curves lies between 500 and 550°C. Varying the amount of the alloying element does not change this temperature appreciably. Below 450°C no evidence is found of gamma-phase transformation until the elapse of considerable time; when the final product is ordered gamma (γ') and alpha in the Mo-U alloys, and niobium-rich (γ_2) phase plus alpha for the U-Nb alloys.

The elevated temperature transformation of the gamma phase for a U-Mo binary alloy is accelerated by the addition of niobium. Molybdenum, on the other hand, when added to a U-Nb binary alloy retards the decomposition of the gamma phase.

Alloying of U-Mo binary with zirconium hastens the transformation of the gamma phase. Since ZrMo_2 is formed, the amount of effective stabilizing Mo is reduced; this is reflected in the shorter transformation time. On the other hand, increasing the amount of zirconium in a U-Nb-Zr alloy retards the high-temperature decomposition of the gamma phase. As the temperature of heat treatment falls from 450°C to room temperature, zirconium has an accelerating effect on the transformation and a new nose appears at about 350°C; from here to room temperature the reaction slows down. Increase in the amount of niobium reduces the rate of transformation at the lower nose, but the zirconium content up to 6 w/o has slight effect on the nose position.

In all of the alloys the transformation at the elevated temperatures is of the cellular or nodular type resulting in a lamellar product. The transformations are not rapid and produce very slight lattice stresses as seen by the small hardening effect. The low-temperature transformation, common to the zirconium bearing alloys, is very rapid and produces great hardening. It appears that it is martensitic in nature; the M_s temperature occurring at approximately 470°C.

It is suggested that the alloys with zirconium will be less corrosion resistant than the U-Mo or U-Nb gamma-phase binary alloys. On the other hand, there are advantages in obtaining high strengths through heat treatment. The U-7.5 Nb-2.5 Zr alloy is attractive for this reason.

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 21. Gamma-quenched from 900°C (400×).

Fig. 22. Gamma-quenched and transformed 8 hours at 600°C (400×).

Fig. 23. Gamma-quenched and transformed 8 hours at 600°C (750×).

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 24. Gamma-quenched and transformed at 550°C for 1 hour (400X).

Fig. 25. Gamma-quenched and transformed at 550°C for 8 hours (400X).

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 26. Gamma-quenched and transformed at 500°C for 0.5 hour (400X, P. L.).

Fig. 27. Gamma-quenched and transformed at 500°C for 1 hour (400X, B. F.).

Fig. 28. Gamma-quenched and transformed at 500°C for 1 hour (400X, P. L.).

Fig. 29. Gamma-quenched and transformed at 500°C for 8 hours (400X, B. F.).

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 30. Gamma-quenched and transformed at 450°C for 1 hour (400X, B. F.).

Fig. 31. Gamma-quenched and transformed at 450°C for 8 hours (400X, B. F.).

Fig. 32. Gamma-quenched and transformed at 450°C for 8 hours (400X, P. L.).

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 33. Gamma-quenched and transformed at 410°C for 0.5 hour (400X, B. F.).

Fig. 34. Gamma-quenched and transformed at 410°C for 1 hour (400X, B. F.).

Fig. 35. Gamma-quenched and transformed at 410°C for 1 hour (400X, P. L.).

Fig. 36. Gamma-quenched and transformed at 410°C for 8 hours (400X, P. L.).

Transformation of gamma-phase 10 Nb-1 Zr-U alloy

Fig. 37. Gamma-quenched and transformed at 350°C for 1 hour (400X, P. L.).

Fig. 38. Gamma-quenched and transformed at 350°C for 8 hours (400X, P. L.).

Fig. 39. Isothermal transformation time versus diamond pyramid hardness for 10 Nb-1 Zr-U alloy.

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