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EFFECTS OF ALLOYING ELEMENT ADDITIONS OF MOLYBDENUM,
SILICON, ALUMINUM, TITANIUM, VANADIUM, AND NIOBIUM
ON THE THERMAL CYCLING STABILITY OF URANIUM

by

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EFFECTS OF ALLOYING ELEMENT ADDITIONS OF MOLYBDENUM, SILICON, ALUMINUM, TITANIUM, VANADIUM, AND NIOBIUM ON THE THERMAL CYCLING STABILITY OF URANIUM

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INTRODUCTION

In an investigation in 1949 on the dimensional stability of uranium on thermal cycling, Kelman and Zegler noted that uranium rods containing 4.5 w/o molybdenum were completely stable to growth and surface roughening after 1500 cycles between 100° and 550°C. The fabrication history of these rods was questionable, but they were believed to have been gamma rolled, heated for 2 hours at 850°C, and water quenched. The rods had a fine-grained structure and shortened 1.47% on annealing 2 hours at 575°C prior to thermal cycling. These observations led to the investigations in this report to explore broadly the effects of molybdenum additions as well as other small alloying element additions on the dimensional stability behavior of uranium during thermal cycling. The alloying elements studied and their nominal concentrations were as follows: Mo, 0.05 - 6.0 w/o; Si, 0.08 w/o; Al, 0.65 w/o; Ti, 0.2 and 0.4 w/o; V, 0.1 and 0.3 w/o; Nb, 0.6 w/o.

MATERIALS

ANL Alloys

The compositions of the binary alloys prepared at ANL for this study are summarized in Table I; their casting and fabrication are described in detail in ANL-5341.(3) The alloys were melted in evacuated induction furnaces, mostly in graphite crucibles; the 6 w/o Mo alloy was melted in a BeO crucible, and the Al, Si, and V alloys in MgO crucibles ("Magnorite"). The melts were bottom poured into graphite or water-cooled copper molds. The castings were in the form of 1-1/2" diameter rods, except for some of the Mo alloys which were cast into 4-1/8" molds. The bulk of the castings were made from remelted project grade slugs of moderately high carbon contents (500-700 ppm); some castings were made from biscuit metal.

The 4-1/8" diameter uranium-molybdenum billets were rolled down to 1-7/16" in diameter at the Simonds Saw and Steel Company, at the nominal rolling temperatures shown in Table I. The castings were pretreated in gas- or oil-fired furnaces and the rolling interrupted occasionally to prevent overheating. The high Mo alloys (2, 4, and 6 w/o) tended to crack on rolling at the lower temperatures; hence they were preheated and rolled at 800-930°C.

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Rolling from 1-1/2" diameter to 1/2" diameter was done at Argonne at the nominal preheat temperatures shown in Table I. An attempt was made at ANL to maintain rolling temperature by soaking the rods 3-5 minutes in a furnace after each pass, but the control of the rolling temperature was unsatisfactory; for the 600°C and 800°C rolling, the temperature along the lengths of the rods was visibly non-uniform. To improve the temperature control during rolling, some of the alloys prepared in the later stages of the program were rolled from lead baths at 600°C (see Table I).

Alloys Received from KAPL and LASL

In addition to the above binary alloys, one ternary alloy containing 150 ppm vanadium, 175 ppm silicon, and 100 ppm molybdenum was tested. This alloy was obtained from the Knolls Atomic Power Laboratory in the "as cast" and "cast and alpha swaged" conditions. Details of casting and/or swaging temperatures are not known. The chemical and spectrographic analysis of this alloy is included in Table I.

Several alloys received from the Los Alamos Scientific Laboratory were also included in this study. Their nominal compositions were: U-2 w/o Mo and U-0.6 w/o Nb.

The thermal cycling results of the KAPL and Los Alamos alloys are given separately in Appendix A.

THERMAL CYCLING AND HEAT TREATMENTS

The alloys were cycled in the following heat-treated conditions:

A. As-rolled at 600°C and annealed at 575°C. The annealing treatment consisted of heating sections of the 1/2" rolled rods for 2 hours at temperature in an argon atmosphere and then air cooling. The maximum alloy concentrations tested in the as-rolled condition were as follows: 0.79 w/o Mo; 0.07 w/o Si; 0.56 w/o Al; 0.35 w/o Ti; and 0.33 w/o V. An attempt to roll a 1.2 w/o Mo alloy at 600°C was unsuccessful.

B. Quenched from 710-725°C. Sections of the 1/2" rolled rods were heated for 2 minutes in a lead bath in the indicated temperature range and water quenched. Following quenching the specimens were annealed at 575°C as above. The alloys given the above heat treatment contained the following maximum alloying element concentrations: 0.79 w/o Mo; 0.07 w/o Si; 0.56 w/o Al; 0.35 w/o Ti; and 0.33 w/o V.

C. Quenched from 850°C (gamma treated). This heat treatment consisted of heating for 2 hours at 850°C in an argon atmosphere and water quenching, followed by annealing at 575°C as above. This heat

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treatment was applied to the uranium-molybdenum alloy series only, with a maximum molybdenum concentration of 6.52 w/o. At 850°C these alloys are in the gamma-phase region.

For thermal cycling the heat-treated alloys were machined and centerless ground to 0.364" in diameter x 1.5" to 4" long. Cycling was carried out in the induction cycler described by Mayfield and Zegler in ANL-4848.⁽²⁾ The specimens were supported vertically in NaK alloy enclosed in a stainless steel container and cycled between 100°C and 550°C, with a heating time of 5-7 minutes, and a cooling time of 20-25 minutes. All specimens were cycled a total of 700 times and length measurements were made with a hand micrometer after 50, 100, 300, and 700 cycles.

RESULTS

Thermal Cycling Growth

"As-rolled" rods: The uranium-molybdenum, uranium-silicon, and uranium-aluminum alloys all grew* in the direction of rolling when cycled in the "as rolled" condition. The growths ranged from 10.8% in 700 cycles for the 0.79 w/o Mo alloy to 26.6% for the 0.07 w/o Si alloy (Table II). By comparison with the growth values reported by Kelman⁽³⁾ for 600°C rolled unalloyed uranium (8.80-14.48% in 700 cycles), additions of molybdenum, silicon, and aluminum in the concentrations indicated apparently did little to reduce the growth of 600°C rolled material. The total growth in 700 cycles was, in effect, higher in some of the alloys as compared with unalloyed uranium, but no special significance should be attached to this since the control of the rolling temperature was poor, and slightly lower rolling temperatures would result in higher growths.^(3,4,5) Also, the smaller grain size in the alloys may contribute to increased growth.⁽⁴⁾

The growths of the specimens are plotted in Fig. 1 as $\ln\left(\frac{L_N}{L_0}\right)$ vs. N, where N = number of cycles, L_N = length after N cycles, and L_0 = original length. Equations of the form $\ln\left(\frac{L_N}{L_0}\right) = aN + bN^2$ were fitted to these curves by the method of least squares, and the growth coefficients G_t^{**} calculated at several cycling levels, as shown in Table II. In all cases the growth coefficients were higher in the early stages of cycling and decreased as cycling continued. This behavior was also noted by Kelman for 600°C rolled unalloyed uranium, as shown in the growth curve plotted from his data in Fig. 1 (specimens 53, 55, and 57 in Reference 1). That the reduced growth rates after continued cycling are not due to increased experimental

*The term "growth" refers to the elongation in the rolling direction.

** $G_t = \left(\frac{dL_N}{dN}\right) \cdot \frac{1}{L_N} = a + 2bN$

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errors of measurement due to warp and surface wrinkling may be deduced from the fact that a specimen remachined after 1500 cycles also had a substantially lower growth rate (compare specimens 55 and 55B in Reference 3).

The as-rolled titanium and vanadium alloys behaved anomalously on cycling; their longitudinal length changes were small compared with the other as-rolled alloys, and both elongations and shrinkages were observed in duplicate specimens of the same alloy (Table II). The length changes were generally more in line with those observed on the 715-725°C heat-treated alloys (see below), and their surfaces wrinkled in a manner characteristic of beta-treated materials; these observations suggest, therefore, that the anomalous behavior is probably a consequence of local overheating into beta-phase temperatures during rolling, resulting in incomplete or partial transformation to the beta phase. That such circumstances during rolling could lead to this inconsistent behavior is further evidenced by the thermal cycling results of a specimen of the 0.08% silicon alloy (discussed below) which had been held at 700°C for only short periods of time to prevent complete transformation. Under these conditions the specimen either elongated or contracted on cycling, depending upon the length of time held at temperature.

Rods quenched from 710°-725°C: The thermal cycling growth data of the alloys quenched from 710°-725°C are summarized in Table III, and the results are plotted in Fig. 2. The major effect of this heat treatment has been to reduce the substantial growth of the "as rolled" materials to relatively low values. Thus, the maximum dimensional change noted in the heat-treated condition for any of the alloys was a 2.6% contraction in 700 cycles (alloy C-898, Table III), as contrasted with a 21.5% elongation in the same alloy "as rolled". In the 710°-725°C quenched condition, the alloys showed no apparent consistency as regards the variation of the growth rate with N: some of the specimens shrank throughout the 700 cycles, others grew, and still others shrank in the early stages of cycling but the deformations changed to growth in the later stages of cycling (alloys C-832 and C-833 in Fig. 2).

The effect of holding time at the heat-treating temperature was investigated for one alloy only - the 0.07 w/o Si composition (alloy C-868). Specimens 0.45" in diameter x 2" long were first presoaked in a lead bath at 600°C for 5 minutes, then transferred to a bath at 700°C, held for times ranging from 2 to 120 seconds, and water quenched. Following heat treatment, the specimens were machined to 0.364" in diameter x 1.5" long and annealed for 2 hours at 575°C. The thermal cycling results, summarized in Table IV, indicate that by varying the holding time within a narrow critical interval one can obtain material which either grows or contracts slightly on cycling. While a holding time of 5 seconds was insufficient to effectively reduce the growth (25.7% in 700 cycles), a holding time of only

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10 seconds reduced the growth to 1.6%, and a holding time of 20 seconds resulted in a 2.0% shrinkage. Increasing the holding time to 120 seconds showed no consistent trend with reference to the variations in the magnitude of the contractions.

Rods quenched from 850°C (gamma quenched): Gamma heat treatment was investigated for the uranium-molybdenum alloy series only, with the molybdenum content ranging from 0.05 to 6.52 w/o. The results are summarized in Table V and Fig. 3, the latter including also the growth curve of an unalloyed uranium specimen similarly heat treated. With the exception of only one alloy, C-795, the total growth in 700 cycles decreased with molybdenum content from a maximum of 2.86% in the 0.05 w/o alloy to a slight shrinkage of 0.10% in the 6.52 w/o alloy. The reason for the anomalous behavior of alloy C-795, 0.46 w/o Mo, which shrank 1.41% in 700 cycles, is not clear. The growth rates varied with cycling level in the manner shown in Fig. 3. The high molybdenum alloys containing 2 to 6.5 w/o Mo showed practically no growth after 700 cycles. The S shape of some of the $\ln\left(\frac{L_N}{L_0}\right)$ vs. N curves, it should be noted, is not confined to the alloys; a similarly shaped curve was obtained for the gamma-quenched unalloyed uranium.

Macro and Microstructural Features

The surface appearances of the specimens after 700 cycles are shown in Figs. 4-6. In the "as rolled" condition (Fig. 4) the specimens that elongated substantially were striated in the rolling direction - a surface condition also characteristic of alpha-rolled unalloyed uranium. It will be noted in Fig. 4 that the vanadium and titanium alloys showed a type of surface wrinkling characteristic of specimens quenched from 710-725°C (see Fig. 5) rather than the striated appearances characteristic of the 600°C "as-rolled" material, suggesting that these alloys were probably heated into the beta-phase region during rolling, which would account for their erratic thermal cycling behavior discussed above.

All specimens quenched from 710-725°C showed evidence of surface wrinkling, but to a somewhat lesser extent than in unalloyed uranium similarly treated. For comparison purposes there is also included in Fig. 5 the surface appearance of unalloyed uranium specimens similarly heat treated (compare Figs. 5b, c, d, and e with 5a).

In the series of uranium-molybdenum alloys that were quenched from 850°C (gamma treated), a gradual improvement with respect to surface wrinkling was noted with increasing molybdenum content, to the extent that the surfaces of specimens with molybdenum contents greater than 0.46 w/o remained unchanged and essentially smooth throughout the 700-cycle test (Fig. 6).

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Representative microstructures of the specimens quenched from 710-725°C before and after cycling are shown in Fig. 7. In the low alloy concentrations, this heat treatment resulted in coarse alpha grain structures not unlike those of unalloyed uranium similarly heat treated, although there were some indications of a slight decrease in the quenched alpha grain size with increasing alloying element additions. Qualitatively the degree of surface wrinkling generally corresponded with the observed grain structures, specimens having the coarsest alpha grain structure also exhibiting the greatest degree of surface wrinkling. Upon thermal cycling, the large-grained alpha structures tended to break up into subgrains of a size generally smaller than those observed in unalloyed uranium.

The microstructures before and after cycling of the low uranium-molybdenum alloys quenched from 850°C are shown in Fig. 8; the microstructures of the alloys containing the higher concentrations of molybdenum (2.12-6.52 w/o) were not investigated, although from the lack of surface wrinkling one might infer that these were of a generally finer unit grain structure.

Hardness

The hardnesses of the gamma-quenched molybdenum alloys are shown in Table VI. The hardnesses increased with increasing molybdenum concentrations up to and including the 2.12 w/o composition. The two higher molybdenum alloys, 3.91 and 6.52 w/o, showed a decided decrease in hardness, undoubtedly indicating changes in transformation behavior*; the hardnesses of these two alloy compositions were restored on annealing at 575°C following quenching. Thermal cycling had no effect on the hardness values, except in the one alloy of 0.46 w/o molybdenum, where a measurable drop in hardness occurred. The reason for the anomalous behavior of this alloy composition is not clear; it should be noted that this alloy behaved anomalously also in thermal cycling (see Fig. 3).

Densities

The densities of some of the alloys before and after cycling are given in Table VII. The densities were measured by the liquid CCl₄ displacement method. In the "as-rolled and 575°C annealed" condition thermal cycling had no significant effect on the densities. The specimens quenched from 710-725°C, however, decreased in density significantly (0.5 to 1%); the decrease may be indicative of phase changes occurring during cycling.

*The drop in hardness is probably due to the retention of the gamma phase, since it has been shown that the gamma phase can be retained in the uranium-molybdenum system at high molybdenum concentrations.

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Thermal Expansion

The thermal expansion coefficients between 25-100°C for some of the molybdenum alloys are given in Table VIII. The data were obtained on the differential dilatometer described in ANL-5221. In the "as-rolled"* condition the expansion coefficients are relatively isotropic. Quenching from 710-725°C had no significant effect; annealing at 575°C after quenching, however, brought about changes in the expansion characteristics, indicating possible retention of a high-temperature phase followed by a gradual decomposition on annealing to the low-temperature stable phases.

Thermal and Electrical Conductivities

The thermal and electrical conductivities of some of the Si, Al, Ti, and V alloys were determined by Battelle Memorial Institute,** using an unalloyed uranium specimen as a comparison standard. The results are summarized in Table IX. The thermal conductivity measurements were made by the heat-wave method, in which the sample is placed with one end in contact with a source of heat and the time required for the other end to come to a specified temperature is measured. For specimens of approximately the same size, shape, density, and specific heat this method gives good comparative values. The estimated precisions of comparative values are $\pm 2\%$ for the thermal conductivity and $\pm 1\%$ for the electrical conductivity. The estimated absolute accuracies are $\pm 10\%$ for thermal conductivity and $\pm 1\%$ for electrical conductivity. Within these limits of precision it would appear that thermal cycling had no significant effect on the thermal and electrical conductivities of these alloys.

Textures

X-ray texture studies were carried out on some of the alloys before and after cycling, using photographic techniques.[†] The 600°C "as-rolled" uranium-molybdenum alloys (maximum 0.79 w/o Mo) exhibited a texture in which the (002) pole is radial, with a scatter toward the rod axis, and the (110) pole is parallel to the rod axis, with a scatter toward the cross direction. The "as-rolled" higher molybdenum alloys, which had been rolled in the gamma-phase temperature range, exhibited no preferred orientation. The effect of gamma treatment (850°C quench) was to remove the texture of the 600°C rolled rods without coarsening the grain size. In the higher molybdenum concentrations, retained gamma phase was noted in the X-ray patterns, but these samples composed into two phases on annealing at 575°C.

*The rolling temperature, 600°C, is in the $\alpha + \gamma$ phase region; see H. A. Saller and F. A. Rough, "Compilation of U.S. and U.K. Uranium and Thorium Constitutional Diagrams," BMI-1000 (June, 1955).

**Private communication, H. A. Nelson to F. G. Foote, March 24, 1949.

[†] Work carried out prior to installation of X-ray spectrometer at ANL.

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Some of the U-Al, Si, Ti, and V alloys that were quenched from 710°-725°C were examined before and after 700 cycles. No preferred orientation was found in these alloys with the above heat treatment before or after thermal cycling. The X-ray patterns also permitted a qualitative evaluation of the grain sizes, which are summarized in Table XIII.

DISCUSSION AND CONCLUSIONS

In studies on the dimensional stability of uranium on thermal cycling one is generally concerned with two major considerations: one, the changes of length occurring in the rolling direction, whether they be positive or negative; and, two, the wrinkling or roughening of the surfaces. The changes of length in unalloyed uranium have been shown to be related to the texture and the grain size, while the surface roughening has been shown to be primarily a function of the grain size. In unalloyed uranium, the heat treatment that is most effective in randomizing the texture and reducing the growth rates to relatively low values (that is, quenching from beta or gamma phase temperatures) unfortunately results also in a coarse alpha grain structure. The grain size may be controlled to a limited extent by varying the times and temperatures a specimen is held at the elevated temperature, but even under the best conditions of heat treatment (that is, with minimum holding times at the lowest possible temperatures) the quenched alpha grain size is still sufficiently coarse to cause considerable surface roughening.

The results of this study would seem to indicate that alloying additions in suitable amounts offer a means of alleviating the surface roughening problem, but that their role in restricting the longitudinal growth is relatively minor. The 600°C rolled alloy rods grew appreciably in a manner similar to unalloyed uranium, although at somewhat different rates; to restrict their growth, quenching from beta or gamma phase temperatures was necessary, in much the same way as for unalloyed material. In the beta- and gamma-treated conditions some of the alloys shrank slightly as compared with the slight elongations noted in unalloyed uranium similarly treated.

The conclusions that can be derived from this study may be summarized as follows:

1. Alloying element additions of 0.79 w/o Mo, 0.07 w/o Si, 0.56 w/o Al, 0.35 w/o Ti, and 0.33 w/o V were ineffective in eliminating the growth of 600°C rolled rods.
2. The above alloying element additions decreased the surface roughness after quenching from beta-phase or gamma-phase temperatures, but to eliminate surface roughening completely a concentration of about

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2 w/o alloying element was needed in the case of molybdenum; the corresponding minimum amounts of the other alloying elements that would be required to effect a similar improvement were not determined.

3. As cast U - 2 w/o Mo specimens and U - 0.6 w/o Nb specimens showed essentially no longitudinal growth and no surface roughening on thermal cycling.

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APPENDIX ATHERMAL CYCLING OF ALLOYS RECEIVED
FROM LOS ALAMOS AND KAPLLos Alamos Materials

The fabrication history of the Los Alamos test rods is summarized in Table X. Also included are two ANL rods that were tested with the Los Alamos material for comparison purposes. The rods were annealed for 2 hours at 550°C and air cooled prior to cycling. Cycling was performed in the Woosher⁽²⁾ using a "5-minute cold - 5-minute hot - 5-second transfer time" cycle. Measurements were made after 10, 50, 100, and 200 cycles, and the dimensional changes are listed in Table XI. The appearance of all test rods, after 200 cycles, is shown in Fig. 9.

Unalloyed Uranium: As expected, the 300°C rolled rods showed the greatest dimensional changes. The 600°C forged samples grew less; the cast specimens grew little but showed considerable surface roughening.

Uranium-Molybdenum Alloys: The 850°C forged and the "as cast" U - 2 w/o Mo alloy specimens shrank slightly on thermal cycling. The cast sample shrank at about the same rate as the sample forged in two planes. The two forged samples in this group showed considerable shrinkage on 550°C annealing prior to cycling. Comparing the improved appearance of the as cast U-Mo specimens with the normal uranium cast specimens indicates that molybdenum must exert a considerable influence on the as-cast grain size. All of the uranium-molybdenum alloy samples were quite smooth and uniform after 200 cycles.

Uranium-Niobium Alloys: In the uranium-niobium group, the growth values for the two 600°C forged specimens were generally comparable with the unalloyed uranium forged specimens. The cast specimen (4023) in this group showed little dimensional change after 200 cycles; it remained smooth and undistorted. The behavior of the "as cast" specimen was similar to that of the cast uranium-molybdenum specimen.

Ternary Alloy from KAPL

The behavior of the ternary alloy received from KAPL (U + 0.015 w/o V + 0.015 w/o Si + 0.010 w/o Mo) is summarized in Table XII. The specimens of this alloy were cycled 700 times in the induction cyler along with all the other ANL alloy specimens described in the report. In the "alpha-swaged" condition the specimens grew 13.5% in 700 cycles; in the "as-cast" and beta-treated conditions they shrank about 1%. The "as-cast" specimens showed considerable surface roughening after 700 cycles (see Fig. 10).

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Table I. Chemical Analyses and Rolling Temperatures

Casting Number	Nominal Rolling Temperature ⁽⁵⁾ (°C)		w/o Alloying Element	Chemical Analyses in ppm ⁽¹⁾															
	4 inches to 1-1/2 inches	1-1/2 inches to 1/2 inch		C	N	Al	B	Be	Cr	Cu	Fe	Mg	Mn	Na	Ni	Si	Sn	Si	Mo
C-809	540	400	0.05 Mo	545	45	<20	<0.1	<5	<1	<1	50	5	7	<1	<5	20	2	<50	-
C-800	540	400	0.10 Mo	878	<10	20	<0.1	1	<1	1.5	85	3.5	5	<1	<5	20	5	<50	-
C-799	540	600	0.20 Mo	598	<10	20	<0.1	6	<1	3.5	75	3.5	6	<2	<5	30	9	<50	-
C-795	540	600	0.46 Mo	700	<10	20	<0.1	<1	<1	3.5	60	2	5	<1	<5	20	6	<50	-
C-820	-	600*	0.40 Mo	876	33	8.5	<0.1	2.5	2.5	2	35	1.5	5	<2	5	40	<5	<50	-
C-898	-	600*	0.39 Mo	575	44	7	.25	<0.5	3	2	30	1	3	<2	5	15	<5	<50	-
C-894	-	600*	0.79 Mo	770	54	7	0.6	<0.5	3	2	40	2	5	<2	5	15	<5	<50	-
C-808	800	800	2.12 Mo	615	18	20	<0.1	<0.5	<1	<1	45	7.5	7	10	<5	40	-	<50	-
C-810	930	800	3.91 Mo	558	10	20	<0.1	<0.5	<1	1	40	2	6	15	<5	40	2	<50	-
C-792	930	800	6.52 Mo	675	53	20	<0.1	50	<1	1	40	1.5	5	10	<5	40	1.5	<50	-
C-868	-	950 and 600 ⁽²⁾	0.07 Si	425	45	25	1.5	<0.5	3	1.5	20	2	3.5	5	20	-	10	<50	<50
C-866	-	800 and 600 ⁽³⁾	0.56 Al	580	28	-	2	<0.5	1	1	30	2	10	7.5	5	15	2	<50	<50
C-833	-	600	0.29 Ti	925	29	<20	<0.1	<0.5	2	<1	150	2	20	<1	<5	70	<1	-	50
C-832	-	600	0.35 Ti	1525	13	<20	<0.1	<0.5	2	<1	50	<0.5	10	2	<5	40	<1	-	<50
C-863	-	600	0.07 V	400	52	20	<0.1	<0.5	2	1	73	2	5	7	<5	30	2	<50	<50
C-869	-	600	0.33 V	475	34	35	<0.1	<0.5	1.5	1	50	2	5	15	5	20	<2	<50	<50
GEV ⁽⁴⁾	(4)	(4)	0.015 V																
			0.017 Si	120	17	20	.3	<0.5	1	1.5	30	2	10	10	25	-	<1	<50	-
			0.010 Mo																

- (1) All analyses are spectrographic except for the added alloying elements, carbon and nitrogen. Analyses are averages of two samples taken from ends and mid-lengths of "as rolled" rods, except for C-820, C-898, and C-894, for which analyses were made on the specimens actually cycled. The following elements were below limits of detection: Ag-<1; Al-<20; As-<10; Bi-<2; Ca-<20; Co-<5; K-<10; Li-<1; P-<20; Pb-<1; Sb-<2; Zn-<20; Ti-<50, except in the Ti alloys.
- (2) The silicon alloy was rolled from the 1-1/2 inch casting to 1-inch round at 950°C and then finish rolled to 1/2 inch round at 600°C.
- (3) The aluminum alloy was rolled from the 1-1/2 inch casting to 1-inch round at 800°C and then finish rolled to 1/2 inch round at 600°C.
- (4) Alloy received from KAPL as 1/2-inch diameter rod in "as cast" and "cast and alpha swaged" conditions; details of swaging temperature not known.
- (5) Alloys noted by* (asterisk) rolled from lead bath; all others preheated in gas furnaces.

Table II. Thermal Cycling Growth Data of Specimens "As Rolled" (600°C) and Annealed at 575°C

Casting Number	Nominal Rolling Temperature (°C)	Specimen Number	Alloying Element (w/o)	% Growth $\left(\frac{L_N-L_0}{L_0}\right) \times 100$				$\ln\left(\frac{L_N}{L_0}\right) \times 10^4$ (experimental)				$\ln\left(\frac{L_N}{L_0}\right) \times 10^4$ (calculated) ⁽³⁾				Constants for Equation ⁽⁴⁾		$G_t^{(2)}$ micro in./in./cycle	
				N = 50	N = 100	N = 300	N = 700	N = 50	N = 100	N = 300	N = 700	N = 50	N = 100	N = 300	N = 700	a x 10 ⁴	b x 10 ⁷	N = 50	N = 700
C-898	600	7-9	0.39 Mo	1.67	3.70	11.4	21.5	166	363	1080	1947	197	384	1044	1918	4.033	-1.848	385	145
		7-10		1.50	3.55	11.1	20.7	149	359	1053	1881								
C-820	600	7-5	0.40 Mo	1.37	3.00	8.4	15.0	136	296	807	1398	156	304	809	1408	3.212	-1.714	304	81
		7-6		1.40	3.12	8.6	15.2	139	307	825	1415								
C-894	600	7-13	0.79 Mo	0.33	1.24	4.9	10.4	33	123	478	989	85	169	481	1010	1.726	-0.4029	169	116
		7-14		0.57	1.58	5.5	10.8	57	157	535	1026								
C-868	950 and 600 ⁽¹⁾	S-1	0.07 Si	3.06	5.00	13.2	26.6	301	488	1240	2359	227	446	1225	2336	4.6430	-1.865	446	334
		S-2		2.61	4.39	12.4	26.1	258	430	1169	2319								
C-866	800 and 600 ⁽¹⁾	A-1	0.56 Al	2.47	3.35	8.0	16.6	244	330	770	1536	152	297	806	1492	3.1054	-0.1391	297	116
		A-2		2.05	3.27	8.1	15.7	203	322	779	1458								
C-833	600	T-3 T-7	0.29 Ti	-0.11 -0.29	-0.18 -0.52	-0.09 -1.20	0.15 -2.67	Not calculated because of anomalous behavior. See text.											
C-832	600	T-11 T-15	0.35 Ti	nil -0.03	0.07 nil	0.24 nil	0.15 0.15												
C-863	600	V-1 V-2	0.07 V	0.42 -0.22	0.78 -0.27	2.11 -0.24	5.15 -1.03												
C-869	600	V-9 V-10	0.33 V	-0.05 nil	-0.05 0.05	-0.05 0.20	-0.20 0.34												

(1) The ingot could not be rolled directly at 600°C, it was found necessary to break up the cast structure by rolling first at 800°C or 950°C as indicated.

(2) $G_t = \left(\frac{dL_N}{dN}\right) \cdot \frac{1}{L_N} = a + 2bN$.

(3) Calculated from equation $\ln \frac{L_N}{L_0} = aN + bN^2$; curve fitted by least squares, averages of the two specimens were used in the calculations.

(4) $\ln\left(\frac{L_N}{L_0}\right) = aN + bN^2$.

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Table III. Thermal Cycling Growth Data of Specimens Quenched from
710-725°C and Annealed at 575°C

Casting Number	Nominal Rolling Temperature (°C)	Specimen Number	Alloying Element (w/o)	% Growth $\left(\frac{L_N - L_0}{L_0}\right) \times 100$				$\ln\left(\frac{L_N}{L_0}\right) \times 10^4$			
				N = 50	N = 100	N = 300	N = 700	N = 50	N = 100	N = 300	N = 700
C-898	600	7-25	0.39 Mo	-0.21	-0.44	-0.93	-2.1	-21	-44	-92	-208
		7-26		-0.21	-0.40	-1.2	-2.6	-21	-40	-119	-257
C-820	600	7-21	0.40 Mo	0.04	-0.09	0.08	0.49	-4	-9	8	49
		7-22		nil	-0.02	0.19	0.65	nil	-2	19	65
C-894	600	7-29	0.79 Mo	0.15	0.33	0.98	1.93	15	33	98	191
		7-30		0.16	0.33	1.01	1.98	16	33	100	196
C-868	950 and 600 ⁽¹⁾	S-5	0.07 Si	-0.22	-0.36	-0.93	-1.77	-22	-36	-92	-176
		S-7		-0.13	-0.29	-0.83	-1.59	-13	-29	-83	-158
C-866	800 and 600 ⁽¹⁾	A-5	0.56 Al	nil	nil	0.10	0.69	nil	nil	10	69
		A-7		nil	nil	0.15	0.34	nil	nil	15	34
C-833	600	T-1	0.29 Ti	-0.07	-0.12	-0.22	-0.10	-7	-12	-22	-10
		T-5		-0.21	-0.21	-0.58	-0.73	-21	-21	-58	-73
C-832	600	T-9	0.35 Ti	-0.11	-0.13	-0.22	-0.20	-11	-13	-22	-20
		T-13		-0.08	-0.15	-0.28	-0.10	-8	-15	-28	-10
C-863	600	V-5	0.07 V	-0.03	0.05	0.20	0.55	-3	5	20	55
		V-6		0.04	0.08	0.33	0.77	4	8	33	77
C-869	600	V-13	0.33 V	nil	nil	0.05	0.06	nil	nil	5	6
		V-15		0.10	0.23	0.74	1.62	10	23	74	161

(1) See footnote (1) in Table II.

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Table IV. Effect of Holding Times at 700°C on Thermal Cycling Growth of U - 0.07 w/o Si Alloy (C-868)

Specimen No.	Holding Time at 700°C(1)	Percent Change in Length $\left(\frac{L_N - L_0}{L_0}\right) \times 100^{(2)}$		
		100 cycles	300 cycles	700 cycles
Si-6	2 seconds; w.q.	4.97	9.30	26.3
Si-7	5 seconds; w.q.	4.42	10.35	25.7
Si-8	10 seconds; w.q.	0.34	0.80	1.6
Si-9	20 seconds; w.q.	-0.24	-0.87	-2.0
Si-10	30 seconds; w.q.	-0.18	-0.04	-2.4
Si-11	60 seconds; w.q.	-0.15	-1.04	-3.1
Si-12	120 seconds; w.q.	-0.15	-0.68	-1.5

(1) All specimens preheated 5 minutes at 600°C before heating at 700°C.

(2) Specimens were originally 0.364" diameter x 1.5" long.

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Table V. Thermal Cycling Growth Data of Rods Quenched from 850°C
and Annealed at 575°C (Gamma Treated)

Casting Number	Nominal Rolling Temperature (°C)	Specimen Number	Alloying Element (w/o)	% Growth $\left(\frac{L_N - L_0}{L_0}\right) \times 100$				$\ln\left(\frac{L_N}{L_0}\right) \times 10^4$			
				N = 50	N = 100	N = 300	N = 700	N = 50	N = 100	N = 300	N = 700
Hanford Slug	600	1-1	-	nil	0.12	0.59	1.07	nil	12	59	106
		1-2	-	0.13	0.31	1.17	N.C.	13	31	116	N.C.
		1-4	-	0.05	0.13	0.60	0.84	5	13	60	84
C-809	540 and 400 ⁽¹⁾	2-1	0.05 Mo	0.09	0.24	1.19	1.57	9	24	118	156
		2-2		0.06	0.15	0.94	N.C.	6	15	94	N.C.
		2-4		0.14	0.30	1.86	2.86	14	30	184	282
C-800	540 and 400 ⁽¹⁾	3-1	0.10 Mo	nil	nil	0.32	1.19	nil	nil	32	118
		3-2		0.03	nil	0.32	N.C.	3	nil	32	N.C.
		3-4		nil	0.07	0.60	0.99	nil	7	60	98
C-799	540 and 600 ⁽¹⁾	4-1	0.20 Mo	-0.03	-0.03	0.05	0.20	-3	-3	5	20
		4-2		0.05	0.06	0.38	N.C.	5	6	38	N.C.
		4-4		nil	nil	0.25	0.54	nil	nil	25	54
C-795	540 and 600 ⁽¹⁾	5-1	0.46 Mo	nil	nil	-0.32	-0.90	nil	nil	-32	-90
		5-2		-0.03	nil	-0.32	-1.15	-3	nil	-32	-114
		5-4		nil	-0.03	-0.45	-1.41	nil	-3	-45	-140
C-808	800	8-1	2.12 Mo	nil	nil	0.13	0.26	nil	nil	13	26
		8-2		nil	nil	0.17	0.37	nil	nil	17	37
		8-4		nil	0.03	0.22	0.35	nil	3	22	35
C-810	930 and 800 ⁽¹⁾	9-1	3.91 Mo	nil	nil	nil	nil	nil	nil	nil	nil
		9-2		nil	nil	nil	nil	nil	nil	nil	nil
		9-4		nil	0.03	nil	-0.03	nil	3	nil	-3
C-792	930 and 800 ⁽¹⁾	10-1	6.52 Mo	nil	nil	nil	-0.09	nil	nil	nil	-9
		10-2		-0.05	nil	-0.04	-0.10	-5	nil	-4	-10
		10-4		-0.03	-0.03	-0.03	-0.08	-3	-3	-3	-8

(1) See Table I for details on rolling temperatures.

N.C. = not cycled.

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Table VI. Hardnesses of Uranium-Molybdenum Alloys (Rockwell A)

Heat Treatment	w/o Molybdenum								
	Unalloyed	0.05	0.10	0.20	0.46	0.79	2.12	3.91	6.52
As rolled ⁽¹⁾	57	58	60	59	62	65	69	57	59
Water quenched from 850°C	57	60	59	61	65	73	69	57	61
Water quenched from 850°C and annealed at 575°C.	54	56	59	59	62	64	67	70	68
After cycling (700 cycles)	54	58	58	59	55	not cycled	66	68	70

(1) See Table I for rolling temperatures.

Table VII. Effect of Thermal Cycling on Densities (grams/cc.)

Casting No.	Alloying Element	As Rolled and Annealed at 575°C			Quenched from 710-725°C and Annealed at 575°C		
		Spec. No.	Before Cycling	After 700 Cycles	Spec. No.	Before Cycling	After 700 Cycles
C-898	0.39 w/o Mo	7-9	18.897	18.888	7-25	18.880	18.745
C-820	0.40 w/o Mo	7-5	18.859	18.854	7-21	18.866	18.761
C-894	0.79 w/o Mo	7-13	18.776	18.771	7-29	18.765	18.578
C-868	0.07 w/o Si	--	--	--	S-8	--	18.794
C-866	0.56 w/o Al	--	--	--	A-8	--	18.103
C-833	0.29 w/o Ti	--	--	--	T-10	18.603	--
					T-14	--	18.607
C-863	0.07 w/o V	--	--	--	V-8	--	18.867
C-869	0.33 w/o V	--	--	--	V-16	--	18.797

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Table IX. Thermal and Electrical Conductivities
(Data supplied by Battelle Memorial Institute)

Casting No.	Alloying Element (w/o)	Specimen No.	Treatment	Per Cent Thermal Conductivity ⁽¹⁾	Per Cent Electrical Conductivity ⁽¹⁾
--	Unalloyed	U-1	Cycled 700x	100	100
--	Unalloyed	U-2	Beta treated and annealed	99.3	99.8
C-868	0.07 Si	S-6	Quenched from 710-725°C and 575°C annealed	94.7	92.4
C-868	0.07 Si	S-7	Same as S-6, cycled 700x	99.3	98.4
C-866	0.56 Al	A-6	Same as S-6	90.9	90.3
C-866	0.56 Al	A-7	Same as S-6, cycled 700x	91.6	92.0
C-833	0.29 Ti	T-2	Same as S-6	97.9	98.4
C-833	0.29 Ti	T-5	Same as S-6, cycled 700x	96.2	98.3
C-863	0.07 V	V-7	Same as S-6	95.8	96.3
C-863	0.07 V	V-5	Same as S-6, cycled 700x	96.2	98.3
C-869	0.33 V	V-14	Same as S-6	96.8	97.8
C-869	0.33 V	V-13	Same as S-6, cycled 700x	99.3	99.4
	Unalloyed	U _A ⁽²⁾	As rolled at 600°C	100	103
	Unalloyed	U _B ⁽²⁾	As rolled at 600°C	102	102.3
	0.005 Si	HSi2 _A ⁽²⁾	As rolled at 600°C	96.8	97.8
	0.005 Si	HSi2 _B ⁽²⁾	As rolled at 600°C	99.3	99.5
	0.15 Si	H6 _A ⁽²⁾	As rolled at 600°C	90.5	88.8
	0.15 Si	H6 _B ⁽²⁾	As rolled at 600°C	91.9	87.8

(1) Thermal and electrical conductivities of U-1 taken as 100% for comparison. Thermal conductivity of U-1 is approximately $0.29 \text{ watt-cm}^{-1}\text{-}^\circ\text{C}^{-1}$ or $0.069 \text{ cal-sec}^{-1}\text{-cm}^{-1}\text{-}^\circ\text{C}^{-1}$ for the range 25 to 120°C. Electrical conductivity of U-1 is approximately $31 \times 10^{-6} \text{ ohm-cm}$ at 26°C.

(2) Specimens prepared by Battelle.

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Table X. Fabrication History of Los Alamos Test Rods

Rod No.	Material	Fabrication Method	Reduction in Area
108	Unalloyed uranium	300°C rolled	75%
1051	Unalloyed uranium	Forged at 600°C, 3 planes, water quenched	87%
1052	Unalloyed uranium	Forged at 600°C, 2 planes, water quenched	87%
1053	Unalloyed uranium	As cast	None
3021	U-2 w/o Mo alloy	Forged at 850°C, 3 planes, water quenched	83%
3022	U-2 w/o Mo alloy	Forged at 850°C, 2 planes, water quenched	83%
3023	U-2 w/o Mo alloy	As cast	None
4021	U-0.6 w/o Nb alloy	Forged at 600°C, 3 planes, water quenched	83%
4022	U-0.6 w/o Nb alloy	Forged at 600°C, 2 planes, water quenched	83%
4023	U-0.6 w/o Nb alloy	As cast	None
4-12*	Unalloyed uranium	300°C rolled and beta treated 1-1/2 minute 700°C, water quenched	89%
4-18*	Unalloyed uranium	300°C rolled	89%

*ANL material

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Table XI. Length Changes of Los Alamos Test Rods on Thermal Cycling Between 50°C and 550°C

Sample No.	% Change After Anneal of 2 hrs. at 550°C	Number of Cycles			
		10	50	100	200
		% Growth $\left(\frac{L_N - L_0}{L_0} \right) \times 100$			
108	+ 0.10	+ 0.42	+ 2.05	+ 3.97	+ 9.02
1051	- 0.01	+ 0.28	+ 1.20	+ 2.38	+ 5.13
1052	+ 0.08	+ 0.28	+ 1.44	+ 2.97	+ 6.40
1053	- 0.02	- 0.05	+ 0.01	+ 0.17	+ 0.52
3021	- 0.24	Nil	- 0.04	- 0.06	- 0.07
3022	- 0.28	- 0.02	- 0.10	- 0.10	- 0.11
3023	- 0.05	- 0.03	- 0.08	- 0.10	- 0.11
4021	+ 0.09	+ 0.27	+ 1.45	+ 2.81	+ 6.17
4022	+ 0.11	+ 0.24	+ 1.41	+ 2.87	+ 6.11
4023	- 0.08	- 0.08	- 0.09	- 0.12	- 0.08
4-12 ⁽¹⁾	+ 0.10 ⁽²⁾	- 0.11	- 0.17	- 0.18	- 0.05
4-18 ⁽¹⁾	+ 1.26 ⁽²⁾	+ 0.50	+ 2.72	+ 5.58	+ 11.83

(1) ANL material

(2) 2 hours - 600°C plus 10 cycles 50°C - 600°C

Table XII. Thermal Cycling Data of Ternary Alloy Received from KAPL
(0.015 w/o V - 0.015 w/o Si - 0.010 w/o Mo)

Specimen Number	Heat Treatment	% Growth $\left(\frac{L_N - L_0}{L_0} \right) \times 100$		
		N = 100	N = 300	N = 700
GEV - 3	Cast and annealed 2 hrs. at 575°C	- 0.16	- 0.40	- 1.04
GEV - 2	Alpha swaged ⁽²⁾ and annealed 2 hrs at 575°C	1.67	4.82	13.5
GEV - 1	Beta treated ⁽¹⁾ and annealed 2 hrs at 575°C	- 0.30	- 0.69	- 1.10

(1) 2 minutes at 725°C and water quenched.

(2) Swaging temperature not known.

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Table XIII X-ray Data of Specimens Quenched from
710-725°C and Annealed at 575°C

Specimen Number	Alloying Element (w/o)	No. of Cycles	Grain Size(1)	X-ray Film No.
U-5	Natural U	700	Coarse	X656
A-8	0.56Al	700	Fine	X648
S-8	0.07Si	700	Medium Fine	X649
T-10	0.35Ti	None	Very Fine	X650
T-14	0.35Ti	700	Medium	X657
V-8	0.07V	700	Medium	X651
V-16	0.33V	700	Medium Fine	X652
GEV-1	V-Si-Mo(2)	None	Fine	X653

(1) As judged from spottiness of X-ray photograms.

(2) Ternary alloy received from KAPL. See Table XII.

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Figure 1

Growth of Specimens "As Rolled" (600°C)

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Figure 2

Growth of Specimens Quenched from 710-725°C

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Figure 3

Growth of Specimens Quenched from 800°C
(Gamma Treated)

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Figure 4. Surfaces of 600°C Rolled and 575°C Annealed Specimens After 700 Cycles.

a. 0.39 Mo (C-898)

Sp. No. 7-9 7-10

b. 0.40 Mo (C-820)

Sp. No. 7-5 7-6

c. 0.79 Mo (C-894)

Sp. No. 7-13 7-14

d. 0.07 Si (C-868)

Sp. No. S-1 S-2

e. 0.56 Al (C-866)

Sp. No. A-1 A-2

f. 0.29 Ti (C-833)

Sp. No. T-3 T-7

g. 0.33 Ti (C-832)

Sp. No. T-11 T-15

h. 0.07 V (C-863)

Sp. No. V-1 V-2

i. 0.33 V (C-869)

Sp. No. V-9 V-10

Macros M-4374, 4413, 4727

1x

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Figure 5. Surfaces of Specimens Quenched from 710-725°C After 700 Cycles.

a. Unalloyed uranium

Spec.# 6U-10 6U-11 6U-12
Macro 12,274 1x

b. 0.39 Mo (C-898)

Spec.# 7-25 7-26
Macro M-4728 1x

c. 0.40 Mo (C-820)

Spec.# 7-21 7-22
Macro M-4728 1x

d. 0.79 Mo (C-894)

Spec.# 7-29 7-30
Macro M-4728 1x

e. Alloying elements as indicated.

T-1	0.29	Ti
T-9	0.35	Ti
S-5	0.07	Si
A-5	0.56	Al
V-5	0.07	V
V-13	0.33	V

Spec.# T-1 T-9 S-5 A-5 V-5 V-13
Macro M-4410 1x

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Figure 6. Surfaces of Specimens Quenched from 850°C and Annealed at 575°C After 700 Cycles.

Macros M-4285 and 4287

1x

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Figure 7. Representative Microstructures of Some of the Specimens Quenched from 710-725°C and Annealed at 575°C Before and After Cycling (700 cycles).

a. 0.07 w/o V alloy before cycling.

Micro 7129

P.L.

150x

b. 0.07 w/o V alloy after cycling.

Micro 6552

P.L.

150x

c. 0.29 w/o Ti alloy before cycling.

Micro 6545

P.L.

150x

d. 0.29 w/o Ti alloy after cycling.

Micro 6548

P.L.

150x

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Figure 8. Representative Microstructures of Uranium-Molybdenum Specimens Quenched from 850°C and Annealed at 575°C Before and After Cycling (700 cycles).

a. 0.05 w/o Mo before cycling.

Micro 6242 P.L. 150x

b. 0.05 w/o Mo after cycling.

Micro 6281 P.L. 150x

c. 0.20 w/o Mo before cycling.

Micro 6258 P.L. 150x

d. 0.20 w/o Mo after cycling.

Micro 6298 P.L. 150x

e. 0.46 w/o Mo before cycling.

Micro 6259 P.L. 150x

f. 0.46 w/o Mo after cycling.

Micro 6300 P.L. 150x

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Figure 9. Appearance of Los Alamos Uranium and Uranium Alloy Test Bars After 200 Cycles Between 50°C and 550°C.

Unalloyed Uranium
(Los Alamos)

U-Mo

U-Nb

Unalloyed
Uranium
(ANL)

Macro M-10171

1-X

Rod numbers left to right:

108	Normal uranium	300°C rolled
1051	Normal uranium	600°C forged, 3 planes
1052	Normal uranium	600°C forged, 2 planes
1053	Normal uranium	"as-cast"
3021	U-Mo alloy	850°C forged, 3 planes
3022	U-Mo alloy	850°C forged, 2 planes
3023	U-Mo alloy	"as-cast"
4021	U-Nb alloy	600°C forged, 3 planes
4022	U-Nb alloy	600°C forged, 2 planes
4023	U-Nb alloy	"as-cast"
4-12	ANL normal U	Beta quenched
4-18	ANL normal U	300°C rolled

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Figure 10. Surface Appearance of Ternary Alloy Specimens Before and After Cycling (700 Cycles).

a. Before cycling

As cast

Macro M-4355

Alpha swaged

Beta-treated

1-X

b. After 700 cycles

As cast

Macro M-4389

Alpha swaged

Beta-treated

1-X