Y-2055

TITANIUM DISTRIBUTION IN URANIUM-TITANIUM ALLOYS

R. L. Ludwig

November 1976

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TITANIUM DISTRIBUTION IN URANIUM-TITANIUM ALLOYS

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ABSTRACT

Electron-beam microprobe analyses were used to assess the uniformity of titanium distribution in both as-cast and heat-treated uranium-0.5 weight percent titanium (U-0.5 Ti) and uranium-0.75 weight percent titanium (U-0.75 Ti) alloys. Microsegregation due to coring was readily smoothed by heat treatment, but long-range variability remained (on the order of 0.1 wt %). An attempt was made to examine titanium macrosegregation by a scanning image analysis measurement of the volume fraction of U_2Ti in an equilibrium microstructure, but a method for consistently obtaining resolvable U_2Ti particles could not be developed.

As judged by tensile properties, a homogenization heat treatment of 1000° C for 24 hours was apparently the most effective of those tried for homogenizing the U-0.75 Ti alloy. The large grain size of the as-cast tensile bars apparently caused variability in the tensile-test results such that it was difficult to closely assess the optimum values of the heat-treatment parameters.

CONTENTS

SUMMARY

Homogeneous dispersal of titanium throughout the uranium-0.75 weight percent titanium (U-0.75 Ti) and uranium-0.5 weight percent titanium (U-0.5 Ti) alloys is essential in assuring uniformity of mechanical properties. A study was undertaken, therefore, to: (1) determine the titanium distribution in these alloys in various conditions of heat treatment, (2) ascertain the effect of heat treatments in smoothing any variability present in the U-0.75 Ti alloy, and (3) study the effect of various homogenization treatments on tensile properties of the U-0.75 Ti alloy.

The electron-beam (EB) microprobe was used for 900-point analyses over 150-μm-square areas in alloys of both compositions. This technique revealed a short-range pattern of dendritic segregation or coring in as-cast alloys which could be significantly leveled by a heat treatment of one hour at 800° C. Application of the microprobe technique showed that cyclic variations in titanium concentration of about 0.1 wt % occurred even after coring had been eliminated.

To find a less time-consuming, yet reliable method for measuring titanium concentration differences, an attempt was made to produce equilibrium microstructures of U_2Ti particles in a uranium solid-solution matrix. An image analyzer was employed to quantitatively measure the volume fraction of U_2Ti in the microstructure. Holding the specimens in the (β + U_2Ti) phase area for a few hundreds of hours produced U_2Ti particles of a measurable size. However, the conditions for consistently producing resolvable particles in all alloy samples could not be isolated. The method appeared limited because of a tendency for the higher titanium contents to cause the high-temperature gamma phase to become increasingly stable.

Homogenization heat treatments for 2 and 24 hours at 800 and 1000° C were run on cast U-0.75 Ti alloy bars. The treatment at 1000° C for 24 hours was superior in that maximum tensile ductility was achieved. The coarse, as-cast grain size and anisotropic crystal structure were believed to be responsible for the low precision in the measurement of properties due to the various heat treatments. As a result, an optimization of the time required for essentially complete homogenization at 1000° C was not obtained.

INTRODUCTION

The mechanical properties of uranium alloyed with 0.5 to 1.0 wt % titanium and properly heat treated considerably exceed those of pure uranium metal. Because of the large effect of small percentages of titanium, it is of considerable importance that it be homogeneously dispersed to ensure isotropy of properties throughout the alloy. A particular problem in the production of cast U-0.5 Ti and U-0.75 Ti alloys has been a variability in tensile properties which could lead to difficulties in adequately certifying the level of these properties within acceptable limits. Although the anisotropic orthorhombic crystal structure of alpha uranium may, in itself (particularly in large grain sizes), lead to a variability among tensile tests, a nonuniform titanium distribution can only accentuate the problem. Therefore, a study was undertaken at the Oak Ridge Y-12 Plant[a] to determine the uniformity of titanium content within the U-0.5 Ti and U-0.75 Ti alloys (particularly the latter) and to ascertain the effect of heat treatments in smoothing any variability present. Also to be studied was the effect of various homogenization heat treatments on the variability in tensile properties of the U-0.75 Ti alloy in both the cast and wrought conditions.

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Energy Research and Development Administration..

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EXPERIMENTAL PROCEDURE

Microsegregation Analysis

Variability in mechanical properties would be expected to be most influenced by relatively long wavelength changes in titanium concentration. That is, variations over distances of the order of the diameter of a tensile bar should be of greater consequence than microsegregation or coring. To completely characterize the as-cast ingot structure and the manner in which it is affected by various heat treatments, an experiment was run in which the EB microprobe was used to measure the microsegregation characteristics of both U-0.5 Ti and U-0.75 Ti alloys. Microprobe analyses were made on a 900-point (30 x 30) grid with the points on 4 to 5-μm centers; and, for some specimens, also on four 9-point (3 x 3) grids spaced at a distance from the large grid. The analysis layout, seen in Figure 1, was designed to reveal both short and long-range concentration variations existing in a given sample. An actual pattern produced by a 900-point microprobe analysis is presented in Figure 2.

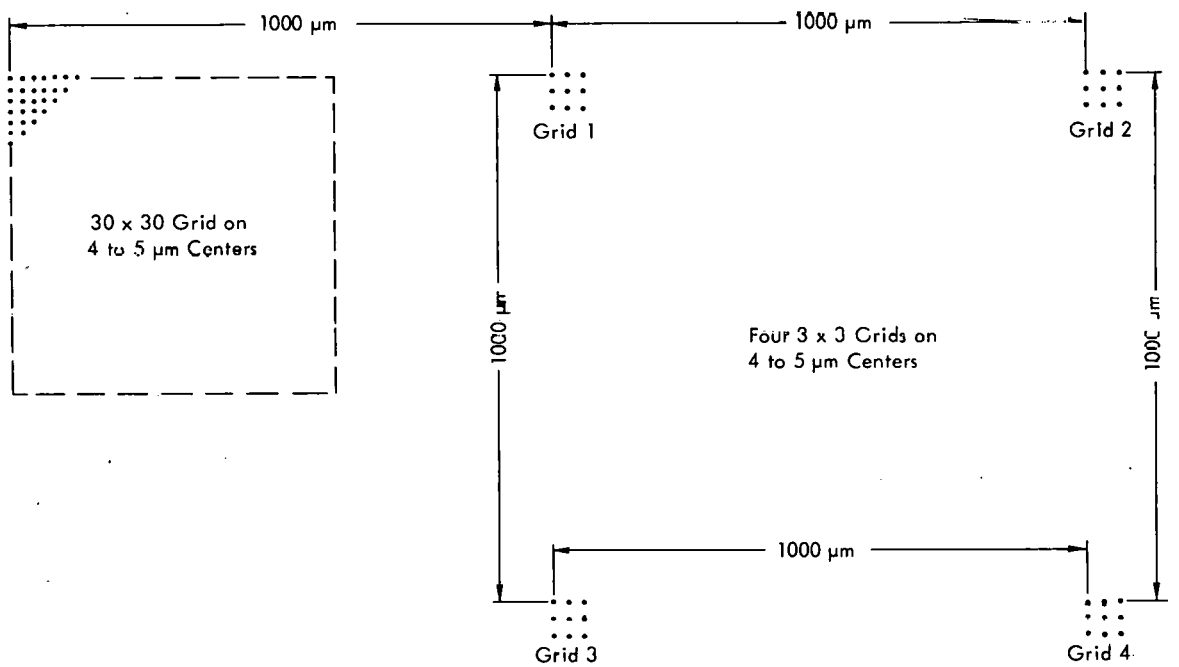

Figure 1. LAYOUT OF SPOT LOCATIONS FOR ELECTRON-BEAM MICROPROBE ANALYSES OF THE URANIUM-TITANIUM ALLOYS.

Figures 3 and 4 are 900-point microprobe analyses of 140 to 150-μm-square areas of nominally U-0.5 Ti and U-0.75 Ti as-cast alloys. The exact origin of the cast U-0.5 Ti sample is not known, but the U-0.75 Ti specimen was cut from the midradius of a 180-mm-diameter vacuum-induction-cast alloy log. Contour lines, roughly delineating the high and low concentration areas, have been drawn in each figure and reveal comparable

segregation patterns. These patterns can be readily visualized as dendritic segregation, with the high-titanium areas being sections of primary dendrites which froze first from the melt. The low-titanium areas are of the lower-melting, titanium-poor liquid which froze last around the dendrites. As would be expected, the highs and lows for the U-0.5 Ti alloy are lower than the corresponding values for the U-0.75 Ti alloy.

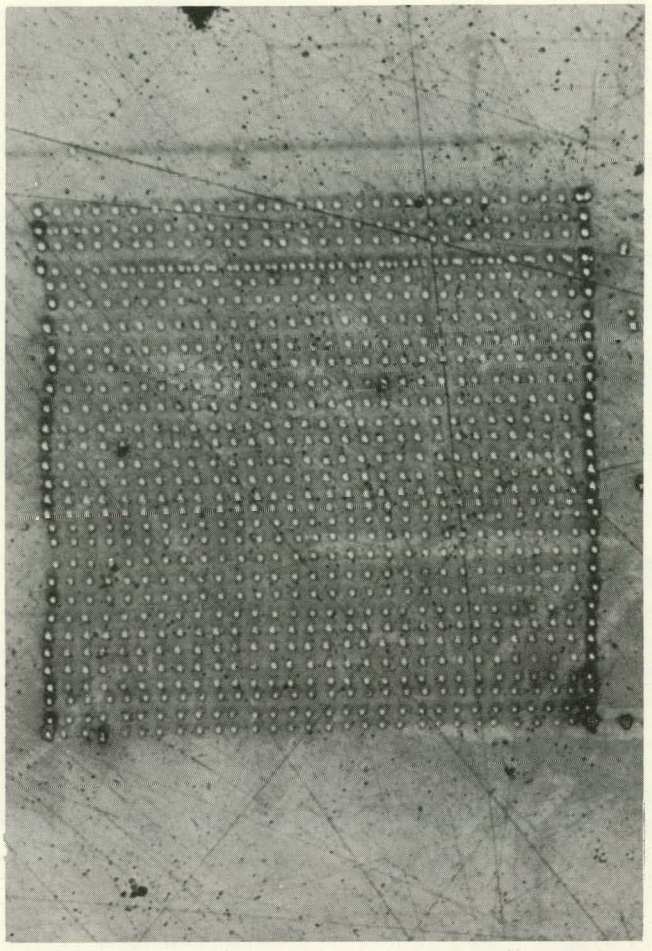

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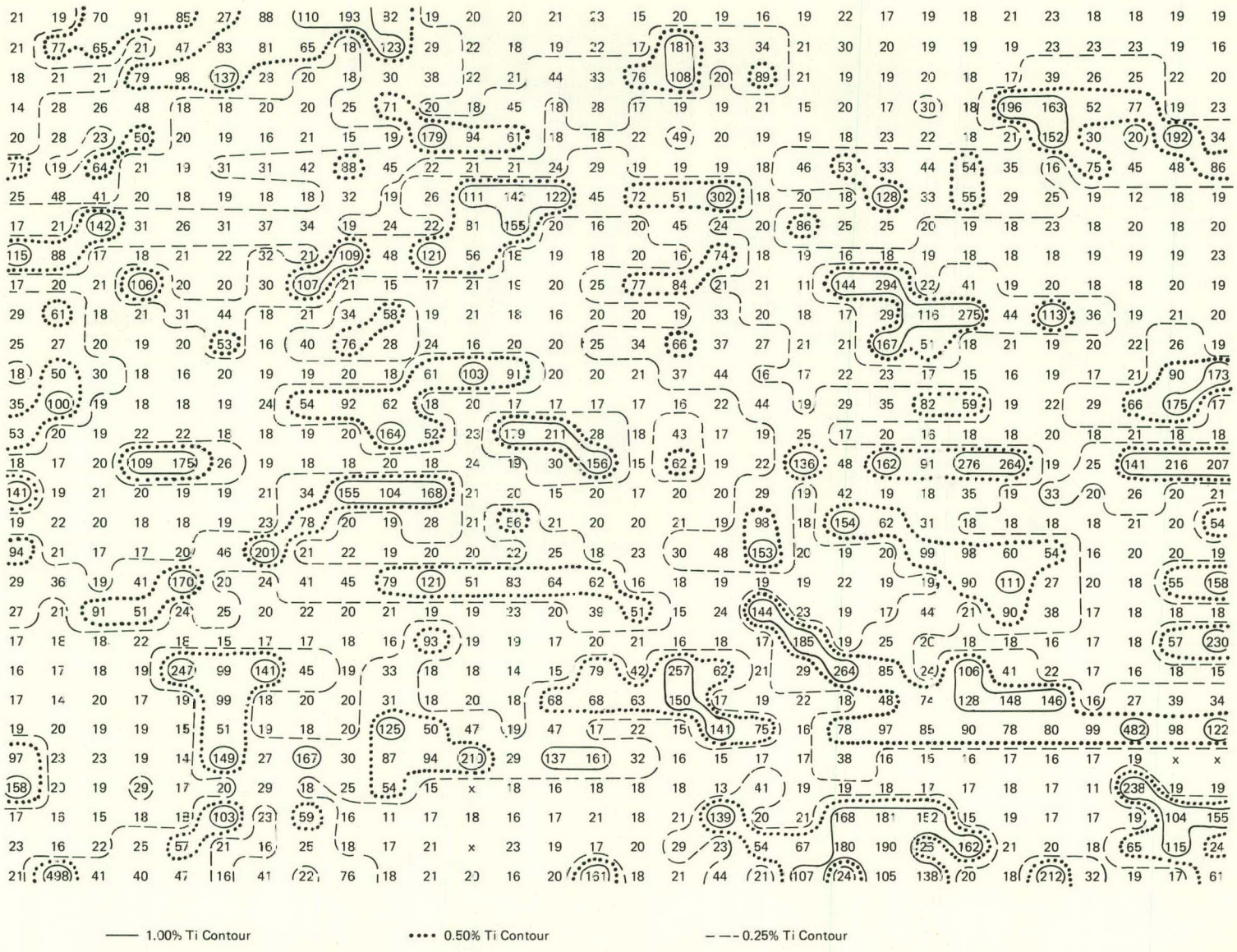
Figure 2. PATTERN REMAINING AFTER A 900-POINT ELECTRON-BEAM MICROPROBE ANALYSIS OF URANIUM-0.75 TITANIUM ALLOY. (Fourth Row from the Top was Inadvertently Scanned Twice; Bright Field Illumination; 500X)

Additional specimens of both alloys from locations adjacent to the first samples were heated at 800° C in vacuum and water quenched. Figures 5 and 6 are plots of the 900-point microprobe analyses made on these specimens. The high-concentration areas have been eliminated, and the titanium gradations in the specimens have been greatly reduced by the diffusion of titanium. Note that in both specimens (Figure 5 in particular), evidence of longer-range gradients exists. That is, the upper half of the area analyzed contains a greater preponderance of higher-concentration areas than the lower half. These differences, however, amount to only a few hundredths of a percent titanium. Another 900-point grid analysis was run for an adjacent U-0.5 Ti alloy specimen which had been aged at 400° C for six hours after receiving the 800° C, one-hour, vacuum-water-quench treatment. No differences existed which can be attributed to the aging treatment. This specimen was similar to that used for Figure 5 in that a longer-range, top-to-bottom titanium gradient appeared to exist.

The influence of a 1000° C, 24-hour, vacuum homogenization treatment followed by the 800° C, 1-hour, vacuum-water-quench treatment is shown for the U-0.75 Ti alloy in Figure 7. This specimen and that of Figure 6 show an average gross difference in titanium of about 0.1 wt %, but were originally no more than 25 to 40 mm apart in the cast log. Aside from this discrepancy, one possible difference between the two appears to be that Figure 7 shows smaller areas of low and high-titanium segregation than Figure 6, a consequence of the long, high-temperature homogenization treatment. The 0.1 wt % titanium difference serves to reinforce the observations made from Figure 5 that longer-range segregation tends to exist. Also, substantiating this premise are the four 9-point grids (Figure 1) associated with Figure 5.



—— 1.00% Ti Contour •••• 0.50% Ti Contour – – – 0.25% Ti Contour

Figure 3. CONTOUR MAP OF ELECTRON-BEAM MICROPROBE TITANIUM ANALYSIS MADE ON 4.8-μm CENTERS FOR AS-CAST URANIUM-0.75 TITANIUM ALLOY. (Numbers are 10^{-2}% Titanium)

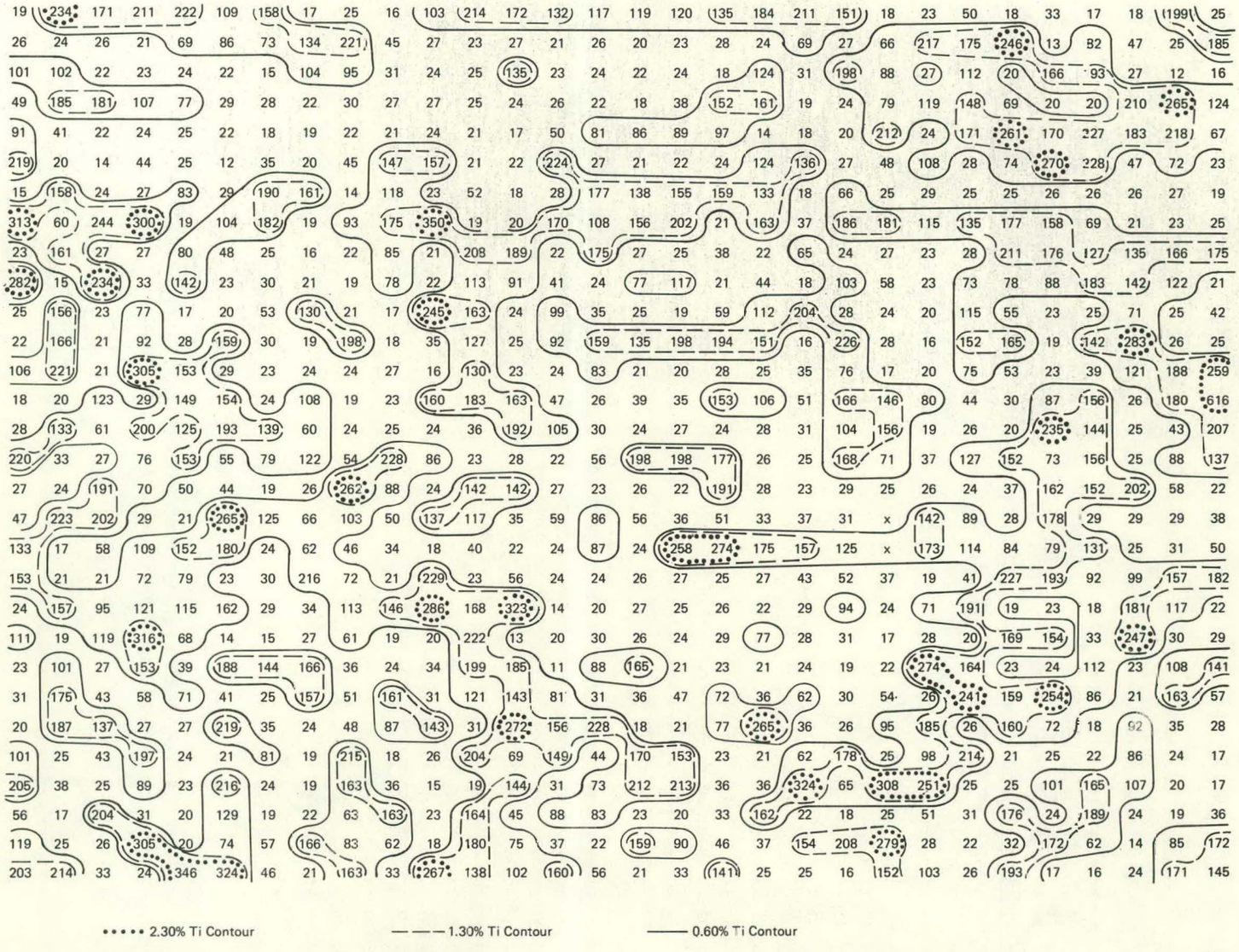


Figure 4. MICROSEGREGATION IN AS-CAST URANIUM-0.75 TITANIUM ALLOY. (Data Points are Spaced Four Micrometers Apart; Numbers are 10^{-2}% Titanium)

——— 0.44% Ti Contour — — — 0.37% Ti Contour

Grid 1

38	37	36
36	44	37
37	37	37

x = 38 + 2

Grid 3

38	34	34
37	36	38
35	37	34

x = 36 ± 1

Grid 2

43	49	47
42	44	40
41	40	42

x = 43 ± 2

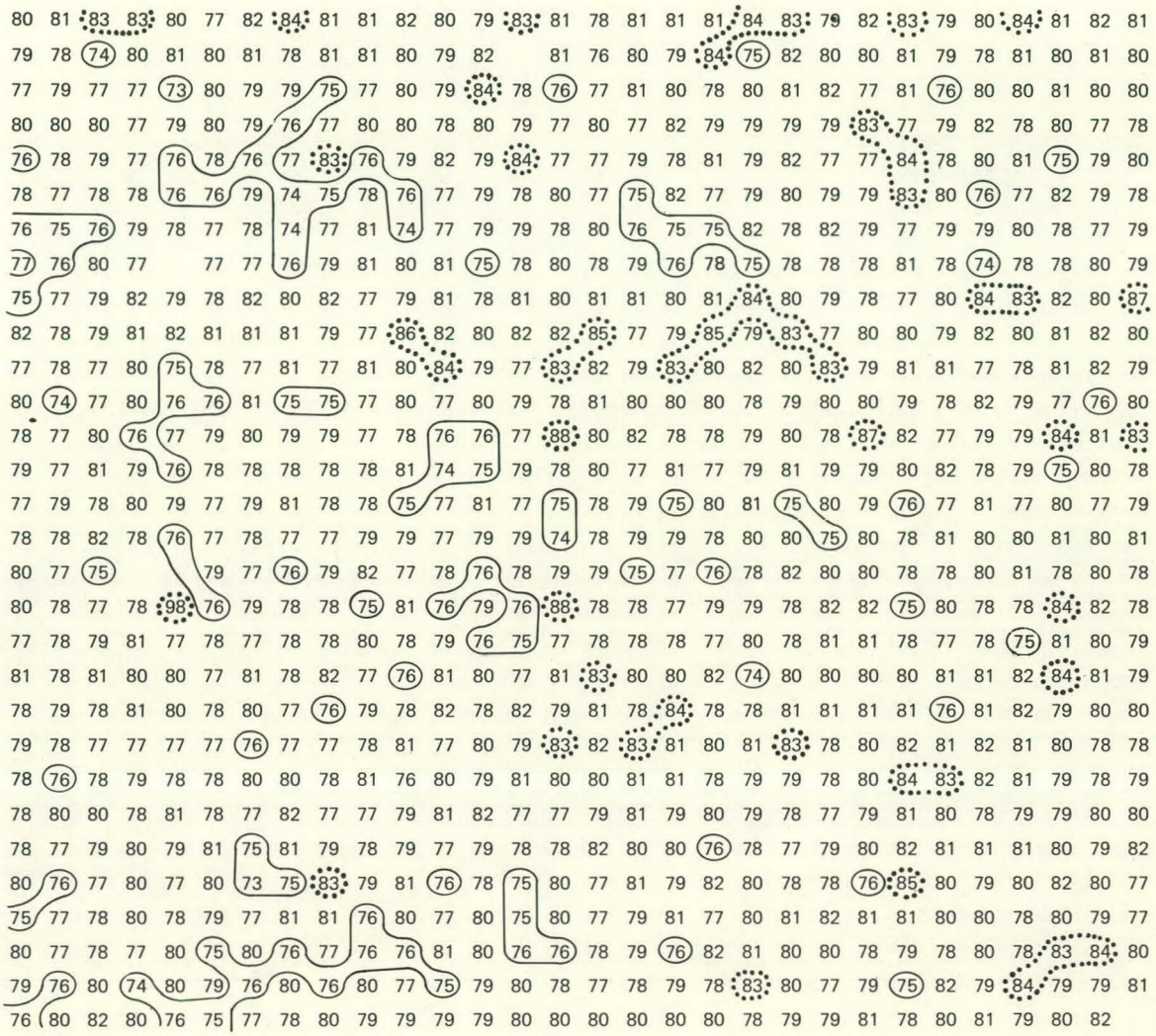
Grid 4

42	42	40
46	44	44
42	42	45

x = 43 ± 1

Four 9 point grids spaced as indicated in Figure 1 with means and 95% confidence limits on the means.

Figure 5. CONTOUR MAP OF THE ELECTRON-BEAM MICROPROBE TITANIUM ANALYSIS ON 4 TO 5-μm CENTERS. (U-0.5 Ti Alloy was Cast and Heat Treated at 800° C for 1 Hour; Average Titanium Analysis—0.40 Wt %; Top Half Average—0.41 Wt %, Bottom Half Average—0.39 Wt %; Numbers are 10^{-2}% Titanium)



••••••0.83 % Ti Contour —— 0.76 % Ti Contour

Figure 6. MICROSEGREGATION IN A URANIUM-0.75 TITANIUM CASTING AFTER ONE HOUR AT 800° C IN VACUUM AND WATER QUENCHED. (Data Points are about 4.5 μm Apart; Numbers are 10^{-2}% Titanium)

In this figure, the titanium concentrations of several of the grids are statistically different from one another and from the 900-point grid.

The tendency for longer-range segregation to occur suggested that the EB microprobe analyses be extended to cover a larger area of the specimen. Accordingly, the specimen from which the data of Figure 7 were obtained was reanalyzed, as outlined in Figure 8, such that an area 900 times as great (4,5 mm square, approaching in size the cross section of a tensile bar) was examined. To achieve this, 100 points on 500-μm centers were analyzed, with the analysis at each point being the average of nine determinations. Figure 9 presents the mean analysis for each location together with the 95% confidence limits on the mean. With only nine analyses per point, most of the points are statistically the same; however, disregarding the limits, there appear to be areas which tend to be somewhat higher or lower than the overall average, but none which approach the titanium level of Figure 6.

– – – 0.73% Ti Contour ——— 0.66% Ti Contour

Figure 7. MICROSEGREGATION IN A URANIUM-0.75 TITANIUM CASTING AFTER 24 HOURS AT 1000° C IN VACUUM, FOLLOWED BY ONE HOUR AT 800° C IN VACUUM AND WATER QUENCHED. (Data Points are about 4.5 μm Apart; Numbers are 10^{-2}% Titanium)

The specimen from which the data of Figure 6 were obtained was analyzed in the same manner as just described, except that, as indicated in Figure 8, the points in the nine-point clusters were on 10-μm rather than 5-μm centers. The data are presented in Figure 10.

The data in Figures 9 and 10, though obtained over a larger area, do not, on the average, appear appreciably different from those in Figures 6 and 7 obtained from small areas of the corresponding specimens. This similarity apparently indicates the existence within a cast ingot of differing compositions which may be relatively uniform, at least over the area of the cross section of a tensile bar. The range of compositions in Figure 10 is greater than in Figure 9, but this difference may be attributable to the homogenization treatment given the specimen from which the data in Figure 9 were obtained.

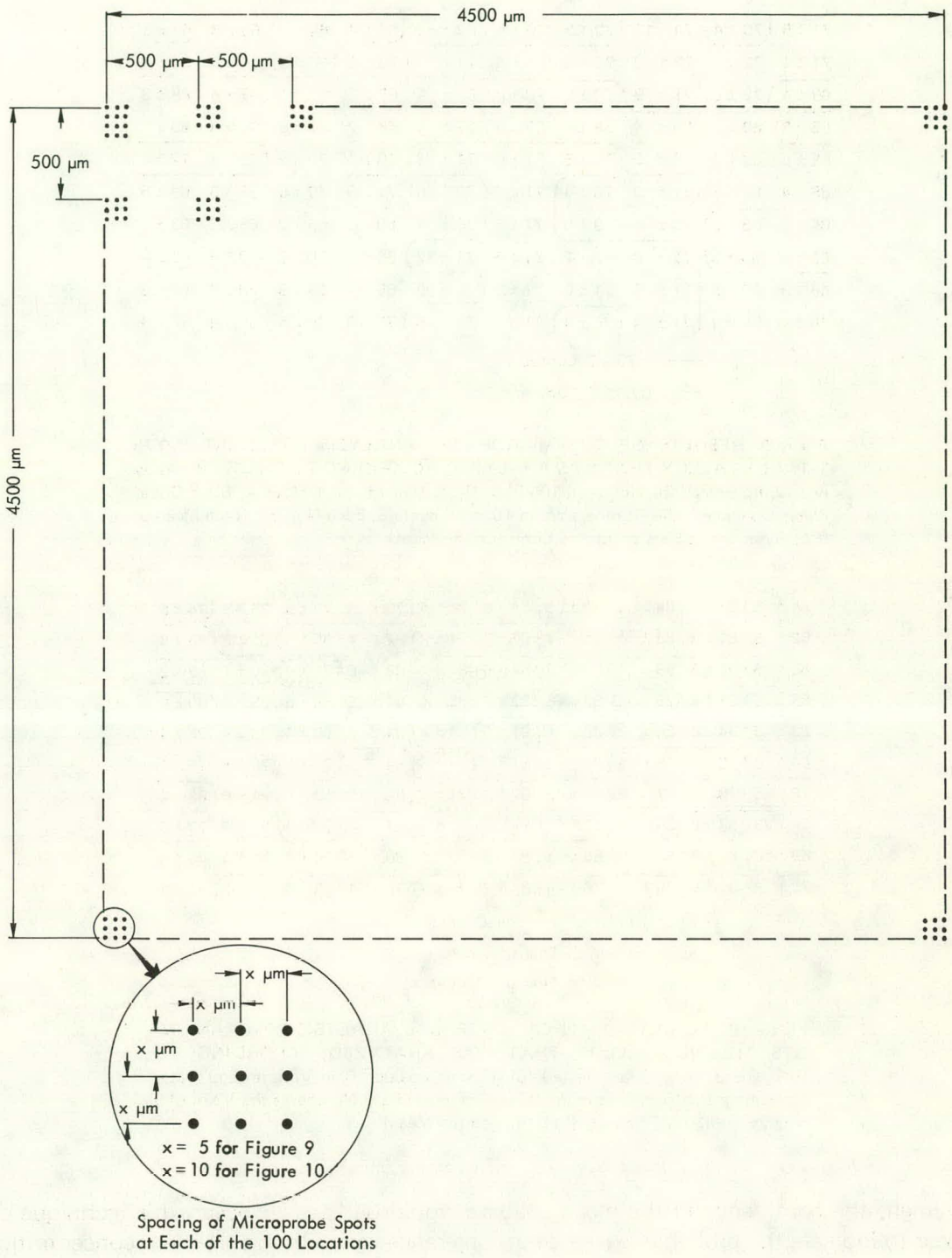


Figure 8. LAYOUT OF ONE HUNDRED 9-POINT ELECTRON-BEAM MICROPROBE ANALYSIS GRIDS ON URANIUM-0.75 TITANIUM ALLOY SPECIMENS.

Attempt to Develop a Method for Quantitatively Measuring Long-Range Titanium Segregation

The microprobe-analysis technique showed the composition of a uranium-titanium alloy specimen only over very small areas; and, unless many analyses were made over a given

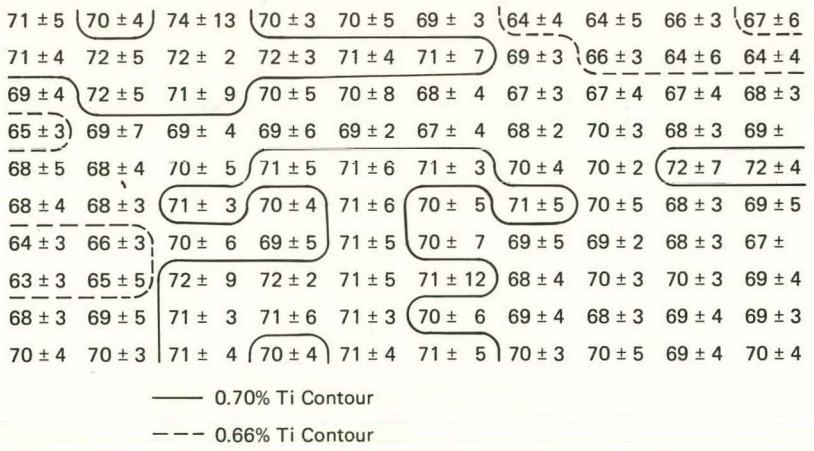


Figure 9. RESULTS OF THE MICROPROBE ANALYSIS OF URANIUM-0.75 TITANIUM ALLOY THAT WAS ANALYZED ACCORDING TO FIGURE 8. (Alloy was Homogenized 24 Hours at 1000° C, then Heated for 1 Hour at 800° C and Water Quenched; the Numbers are in 10^{-2}% Titanium; Each Number is the Mean of 9 Analyses with 95% Confidence Limits on the Mean)

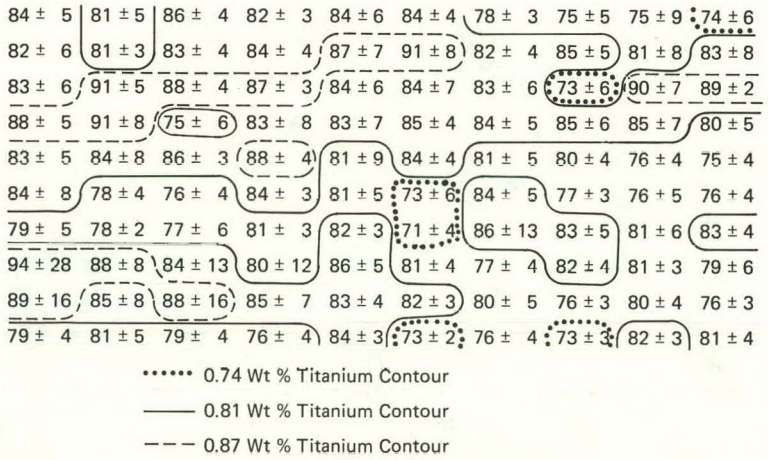


Figure 10. RESULTS OF THE MICROPROBE ANALYSIS OF URANIUM-0.75 TITANIUM ALLOY THAT WAS ANALYZED ACCORDING TO FIGURE 8. (Alloy was Heated for 1 Hour at 800° C in Vacuum and Water Quenched; the Numbers are in 10^{-2}% Titanium; Each Number is the Mean of 9 Analyses with 95% Confidence Limits on the Mean)

specimen, the confidence in the mean analysis would be low. However, this technique did appear to indicate the probable existence of long-range variability in titanium concentration, so it was desirable to seek a means by which the average titanium concentration over relatively large areas of a specimen could be measured. One possible method of accomplishing this goal would be a quantitative metallographic measurement of the intermetallic compound, U_2Ti, existing in an equilibrium microstructure. If phase equilibrium could be achieved, this method would produce valid results since the distance over which titanium variability was of interest was long in comparison with the diffusion distances required to form the U_2Ti particles. In addition, the availability of a Quantimet

image scanning analyzer gave the capability for rapid, accurate volumetric measurements of the U_2Ti, provided the particles were of a resolvable size.

The uranium-titanium phase diagram shows the two components to be mutually soluble in all proportions at elevated temperatures. In the range of compositions of interest in this report, the high-temperature gamma solid solution decomposes eutectoidally (the 0.5 Ti alloy is hypoeutectoid and the 0.75 Ti alloy is approximately a eutectoid composition) at about 723° C into the beta solid solution and U_2Ti. At about 667° C, the beta becomes unstable and reacts either eutectoidally or peritectoidally in combination with U_2Ti to yield an alpha solid solution and U_2Ti, which are the stable room-temperature phases. An effort was made to produce an equilibrium structure by heating in the ($\alpha + U_2Ti$) region of the phase diagram. A specimen was cut from the midradius of a 185-mm-diameter cast U-0.75 Ti ingot, wrapped in tantalum foil, vacuum encapsulated in quartz, and heated at 600° C for 216 hours. At the end of this period the capsule was broken under water to quench the specimen. The resulting microstructure appears in Figure 11. The U_2Ti is a very fine precipitate outlining the alpha solid-solution platelets, much too fine a particle for accurate resolution by the Quantimet at its working magnification.

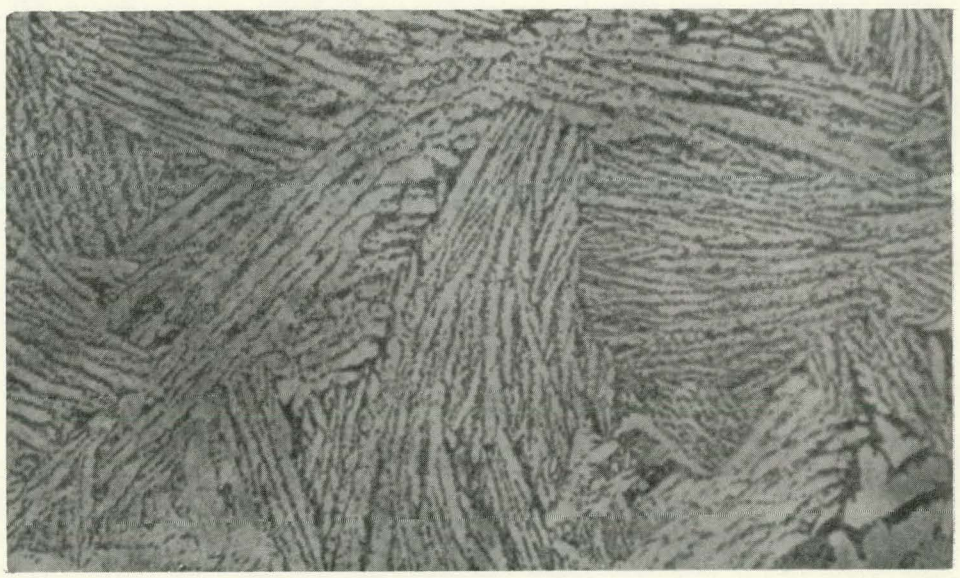


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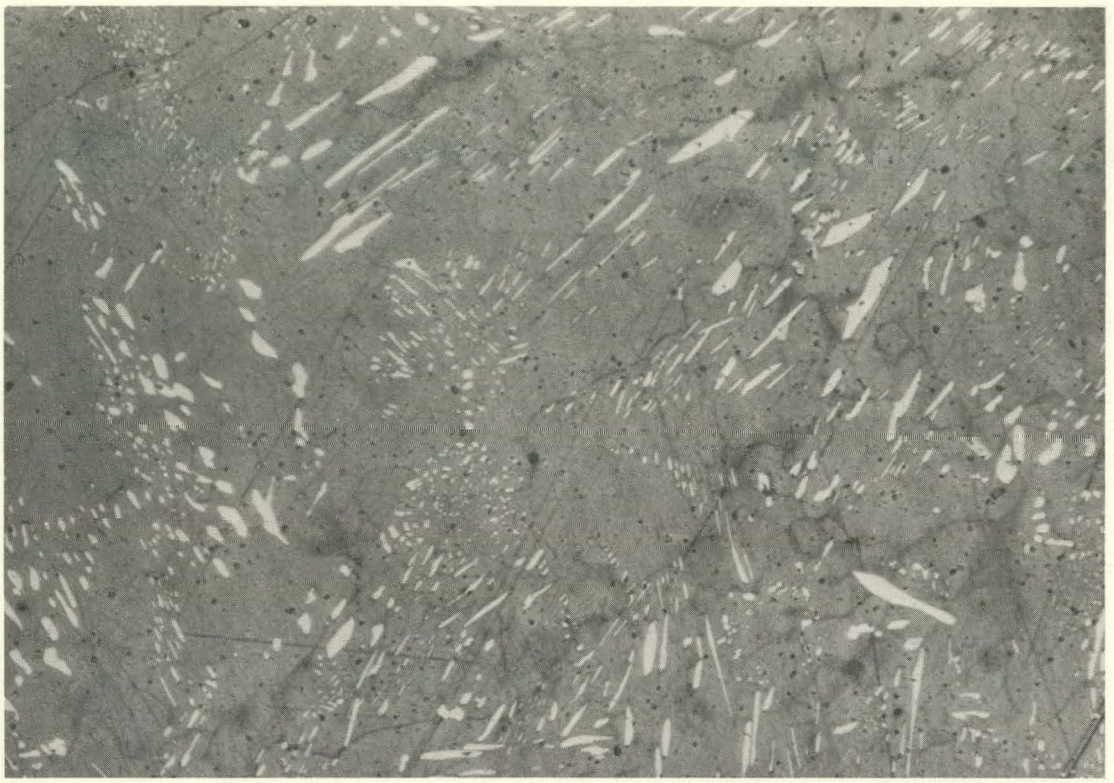
Figure 11. CAST URANIUM-0.75 TITANIUM ALLOY AFTER HEATING FOR 216 HOURS AT 600° C IN VACUUM. (Oxalic Acid Etch; Bright Field Illumination; 1000X)

It was decided that if the equilibrating heat treatment were performed in the ($\beta + U_2Ti$) phase region, the morphology of the U_2Ti particles might be different; ie, larger and more resolvable. A second specimen was taken directly adjacent to the first, encapsulated in the same manner, and heated for 264 hours at 690 - 700° C. The U_2Ti particles were mostly large and resolvable, and showed characteristics of a hypoeutectoid structure, as noted in Figure 12. The structure produced in this specimen was the most satisfactory of any obtained during the entire experiment. As will be pointed out in the discussion to follow, this structure was never closely duplicated despite an extensive effort to do so.

A Quantimet analysis was made of this specimen to measure variations in the volume fraction of U_2Ti present, as indicated by its areal fraction on the polished surface. Two



J186-A

Figure 12. CAST URANIUM-0.75 TITANIUM ALLOY AFTER HEATING FOR 264 HOURS AT 690 - 700° C IN VACUUM. (Air Oxidized; Bright Field Illumination; 500X)

surveys were made, one to show area-to-area differences and the other to reveal the continuous variation along lines on the specimen. The first survey covered 30 points on a grid that was 8 by 6 mm. In the 8-mm direction were five points 2 mm apart, while the six points in the 6-mm direction were 1.2 mm apart. At two random locations on the sample, the second survey was made. At each of these, four continuous, linear traverses (4 mm long) were made in a tick-tack-toe pattern, with the center square 2 mm on a side. The area covered by a Quantimet scan is approximately 0.25 by 0.20 mm, so each point on the 30-point survey covers 0.05 mm^2. For the line-traverse survey, each band was formed by 25 butting or slightly overlapping fields of this size. These data are presented in Tables 1 and 2.

Figure 13 was prepared to show the relationship between the volume percent U_2Ti in the alloy microstructure and the

Table 1

VOLUME PERCENT U_2Ti AT 30 POINTS IN THE MICROSTRUCTURE OF URANIUM-0.75 TITANIUM ALLOY HEATED IN VACUUM AT 695° C FOR 264 HOURS. (Columns are 2 mm Apart and Rows are 1.2 mm Apart; 0.05 mm^2 Area Scanned at Each Point)

Row Number	Column Number 1	2	3	4	5
1	8.22	7.39	7.87	6.84	8.72
2	8.34	10.33	6.92	5.66	7.41
3	6.63	7.04	12.76	6.88	8.79
4	10.95	8.16	10.21	6.00	5.77
5	7.40	7.64	7.91	8.08	6.16
6	8.32	7.66	5.70	5.60	7.05
Overall Average	7.75				

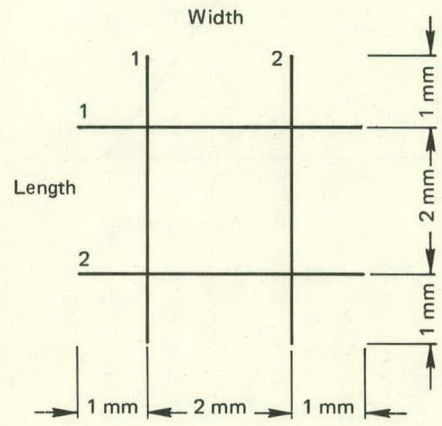
Table 2

VOLUME PERCENT U_2Ti ALONG FOUR CONTINUOUS BANDS AT TWO LOCATIONS ON A URANIUM-0.75 TITANIUM ALLOY SPECIMEN HEATED IN VACUUM AT 695° C FOR 264 HOURS. (Band Pattern at Each Location as Shcwn in the Sketch; Top Numbers in the Columns Begin at the Top and Left of the Lines in the Sketch; Each Number is the Vol % U_2Ti in an Area 1/25 of the Corresponding 0.25 by 4 mm Band)

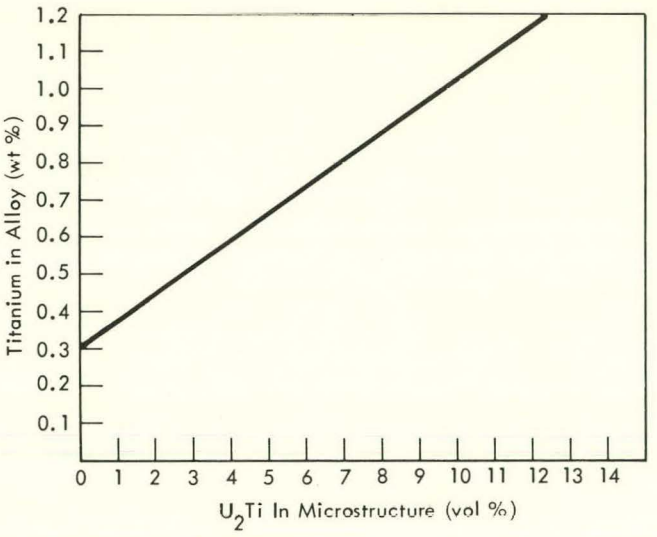
	Location 1				Location 2			
	Width 1	Width 2	Length 1	Length 2	Width 1	Width 2	Length 1	Length 2
	7.68	3.47	7.50	6.51	5.79	7.43	8.01	5.86
	7.48	6.67	5.69	5.43	6.77	5.72	6.36	5.51
	5.78	5.65	4.99	7.79	6.92	8.80	4.20	7.51
	7.17	7.17	10.66	5.61	5.01	7.00	7.32	5.38
	6.87	8.53	7.31	5.46	6.51	5.19	4.83	7.26
	8.94	8.38	7.55	6.30	5.18	5.66	8.07	6.87
	7.36	7.77	5.31	7.29	5.50	7.24	6.35	6.49
	9.61	7.98	5.35	7.10	5.55	6.82	5.96	6.41
	6.64	7.24	7.07	6.70	4.66	4.94	10.07	5.82
	7.04	7.20	6.45	6.05	6.09	4.47	6.37	5.58
	7.48	6.80	6.68	7.70	5.22	5.40	6.59	6.84
	6.59	7.39	8.02	6.17	5.90	4.02	6.64	8.12
	6.51	6.61	6.03	6.52	4.83	4.81	7.13	6.66
	6.58	6.57	6.79	6.73	7.55	4.54	6.28	7.52
	6.32	5.98	6.01	7.68	5.40	6.45	6.64	7.69
	5.65	5.89	5.29	8.07	6.33	6.83	6.09	6.12
	6.59	6.17	4.66	6.59	6.81	6.83	7.74	6.85
	6.05	7.50	5.04	5.93	6.46	6.87	5.04	6.20
	8.28	5.65	8.55	9.24	5.99	10.01	7.14	10.31
	7.56	8.01	7.82	8.50	5.57	8.99	8.85	7.53
	7.76	6.40	8.22	8.31	7.63	8.11	9.63	7.36
	7.40	7.87	6.10	5.47	6.88	7.33	5.65	8.60
	6.61	7.39	6.46	9.25	7.33	7.02	6.55	6.08
	6.23	7.12	6.74	6.58	6.68	5.57	6.99	8.30
	5.53	4.83	9.17	7.61	7.60	8.99	8.21	7.29
Average	7.03	6.81	6.78	6.98	6.17	6.60	6.91	6.97

Average for Location 1 = 6.90

Average for Location 2 = 6.66



weight percent titanium in the alloy. To perform the calculation, a solubility of titanium in the beta solid solution at 695° C was assumed. A value of 0.3 wt % was selected from the literature.(1) Using this figure, the data in Tables 1 and 2 may be close to being quantitative, but probably show too high a titanium content. An attempt was made to ascertain if the data in Table 2 showed cyclic variations in titanium content of a near-constant wavelength. The data for each of the bands were plotted; and, although there was considerable scatter, some cyclic variation appeared to be present. These variations became more evident when, rather than plotting each data point separately, the averages of each two successive points were plotted. Figure 14 shows the plots of half of the data of Table 2 which smooth curves sketched in.



Sample Calculation:

Basis: 100 cm^3 of Alloy for 7 vol % U_2Ti

Weight U_2Ti = 106.4 g

Weight U in solid solution = 1756.5 g

Weight Ti in U_2Ti = 9.73 g

Weight Ti in solid solution = 5.27 g

Total Ti = 15.00 g

$$\text{Weight \% Ti} = \frac{15.00}{106.4 + 1756.5} (100) = 0.805$$

Figure 13. VOLUME PERCENT U_2Ti IN THE MICROSTRUCTURE VERSUS THE WEIGHT PERCENT TITANIUM IN THE ALLOY, ASSUMING 0.3 WEIGHT PERCENT TITANIUM IN SOLUTION. (Density of U_2Ti, 15.2 g/cm^3; Density of U-0.3 Ti, 18.89 g/cm^3)

In view of the apparent success of the transformation heat treatment in yielding an essentially equilibrium structure which could be used to assess the titanium content, the experiment was repeated to determine the effect of homogenization on titanium distribution. Small specimens were cut from near the center of a 185-mm-diameter cast ingot of nominal U-0.75 Ti alloy. One specimen was heated to 800° C for one hour in vacuum and water quenched; the other specimen was heated at 1000° C for 24 hours in vacuum and furnace cooled. Both specimens were wrapped in tantalum foil and vacuum encapsulated in separate quartz capsules and heated at what was believed to be 680 - 700° C for 264 hours. However, metallographic examination revealed that the furnace temperature must have been nonuniform. The 800° C specimen appeared to have been heated above 723° C because of the normal acicular structure which would be caused by rapid cooling from above the eutectoid temperature; the 1000° C specimen appears to have partially transformed at a slightly lower temperature, as shown in Figure 15. The structure is shown not necessarily because it adds to the homogenization study, but because it may contribute to an understanding of the transformation kinetics. From the figure it appears evident the hypoeutectoid islands of beta formed first until the surrounding residual gamma became of the eutectoid composition which then transformed into (β + U_2Ti), with the U_2Ti particles being very large. To get an idea of the partition of titanium among these three phases, an EB microprobe analysis was run. As suspected, the white needles were U_2Ti, containing 8.77 ± 0.23 wt % (32.07 ± 0.69 at %) titanium; the light, formerly beta phase, was 0.29 ± 0.06 wt %, and the dark, formerly gamma phase, analyzed 1.27 ± 0.19 wt % titanium.

The fact that the transformation of this specimen was incomplete, while the one discussed just previously (Figure 12) was completely transformed in the same length of time indicates that there may have been influential factors which had not been considered. The 1000° C homogenization treatment was a difference between these two specimens and suggests that a specimen pre-treatment which completely disperses the titanium may be important. However, the transformation temperature may also have been significantly different, so an experiment was run to examine this variable. Specimens about 14 by 14 by 6 mm were cut from the midradius of the casting from which the previous specimen was obtained. The specimens were not heat treated in any way and were wrapped in tantalum and vacuum sealed in quartz. The ($\beta + U_2Ti$) phase region reportedly ranges from 667 to 723° C, so three transformation temperatures within this range—675, 695, and 715° C—were selected as the values for the controlled variable in this experiment. The temperatures were controlled to ± 5° C by a calibrated

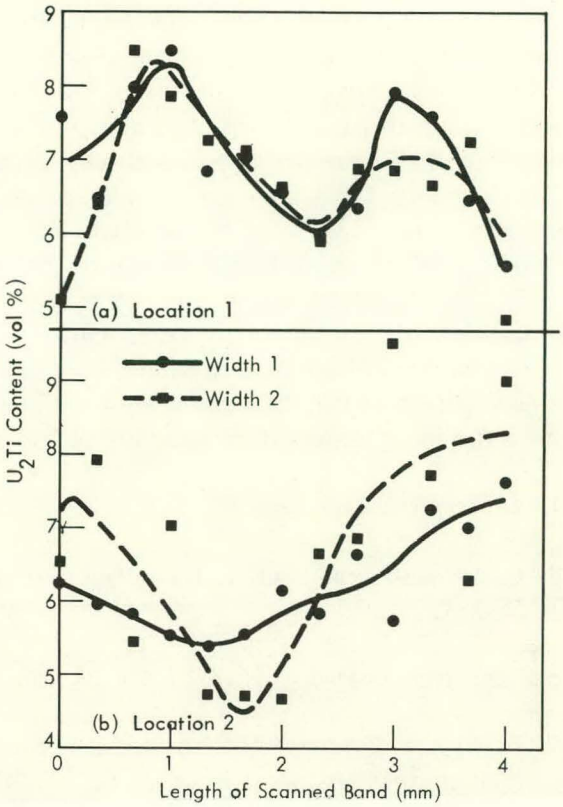


Figure 14. CYCLIC VARIATIONS IN VOLUME PERCENT U_2Ti ALONG THE QUANTIMET SCANS ACROSS URANIUM-0.75 TITANIUM ALLOY THAT WAS HEATED IN VACUUM AT 695° C for 264 HOURS.

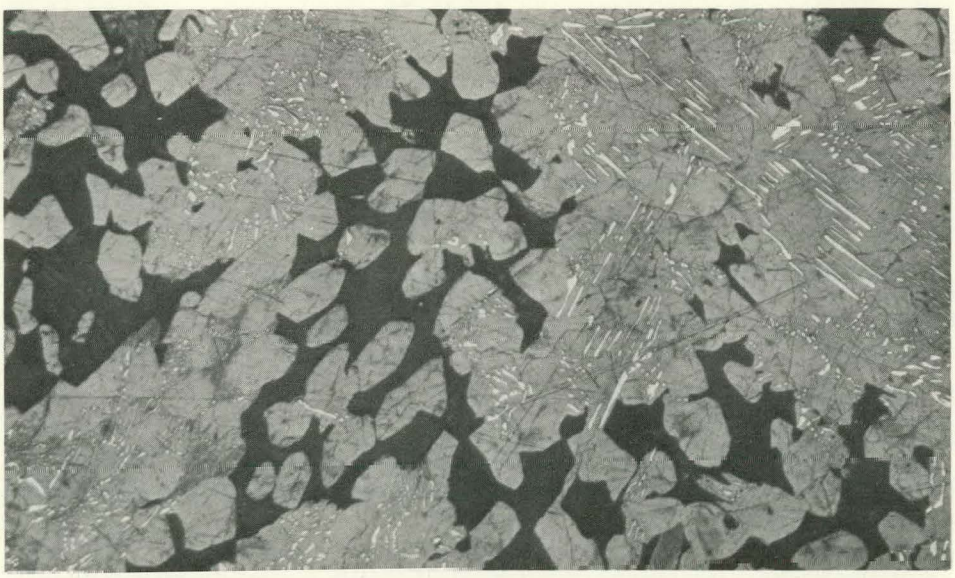


Figure 15. CAST URANIUM-0.75 TITANIUM ALLOY THAT WAS HOMOGENIZED AT 1000° C IN VACUUM FOR 264 HOURS. [Partially Transformed in the ($\beta + U_2Ti$) Phase Region; Air Oxidized; Bright Field Illumination; 250X]

thermocouple immediately adjacent to the specimen. A transformation time of 72 hours was selected for a first attempt. Figure 16 consists of photomicrographs of the three transformed specimens. All three are apparently completely transformed in that the background phase is continuous and they show areas of no U_2Ti, indicating hypoeutectoid behavior. In Figure 16, Views a and c, in particular, much of the U_2Ti is too fine, even at 1000X, to be resolvable and so would not be amenable to quantitative measurement by image analysis. The fine U_2Ti particle size obtained in the preceding experiment suggested that longer times at temperatures might lead to coalescence and growth, and that the 695° C temperature was as effective as any in effecting the transformation. Also, the results obtained with the homogenized specimen (Figure 12) indicated that the influence of prior heat treatment should be studied. Therefore, heat-treated specimens were transformed for a longer period at 695° C. The specimens were from the same U-0.75 Ti alloy ingot as those from the three-temperature experiment and were treated as follows:

1. No treatment—as cast.

2. Cast, heated at 800° C for 1 hour in vacuum, water quenched, and aged 6 hours at 380° C in vacuum.

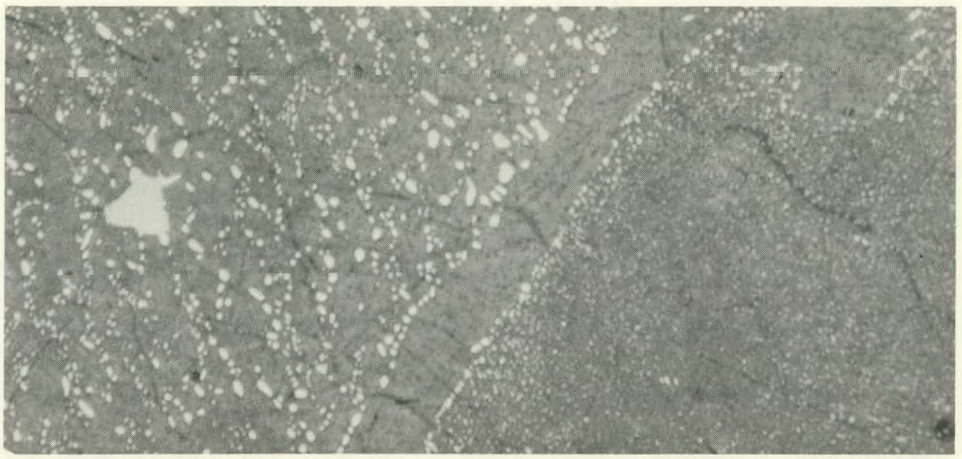
3. Cast, then heated at 1000° C for 24 hours in vacuum.

4. Rolled at about 625° C to 55% reduction, heated at 1000° C for 24 hours in vacuum, heated at 800° C for 1 hour in vacuum, and aged 6 hours at 380° C in vacuum.

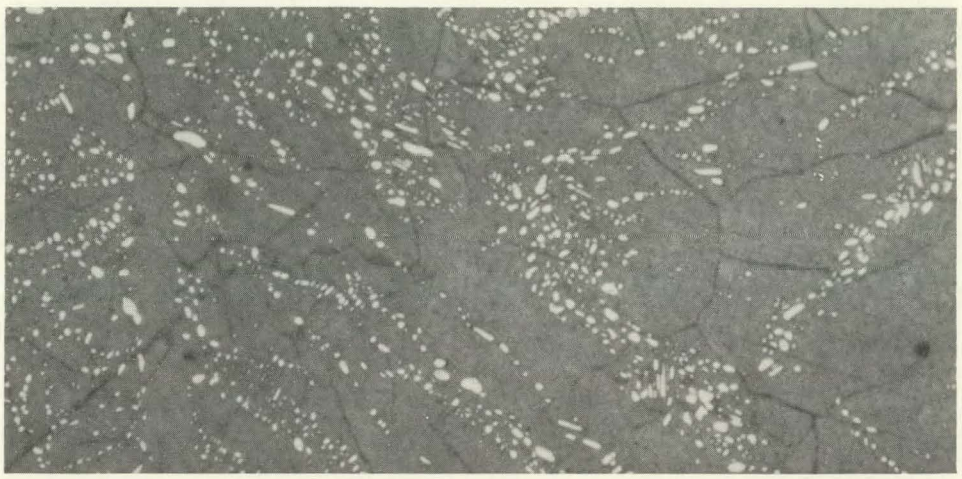
All were wrapped in tantalum and vacuum encapsulated in quartz, Specimen 1 in one capsule and Specimens 2, 3, and 4 in another. The two capsules were loaded into the furnace with the specimens adjacent to the calibrated thermocouple for accurate temperature control and maintained at 695 ± 5° C for 208 hours. Figure 17 shows the microstructures of the as-cast specimen at two magnifications. The extended time at temperature apparently had little effect on the U_2Ti particle size except possibly to slightly enlarge the finest particles. In View a, the dark areas appear to be regions of high titanium concentration, as suggested by the dense population of small particles in View b. The higher titanium may have caused the precipitation and growth reaction to be sluggish in these areas. Figure 18 shows the microstructures of the other three specimens. The heat treating and working appears to have resulted in a more uniform distribution of the U_2Ti, but there is little or no improvement with respect to particle size.

With the failure of the increased time to cause the formation of a larger U_2Ti particle size, an as-cast U-0.75 Ti alloy specimen, wrapped in tantalum and encapsulated in quartz, was heated at 675 ± 5° C for 425 hours. The microstructure of this specimen was much the same as in View b of Figure 17, indicating that prolonged heating is not the primary factor in increasing the U_2Ti particle size.

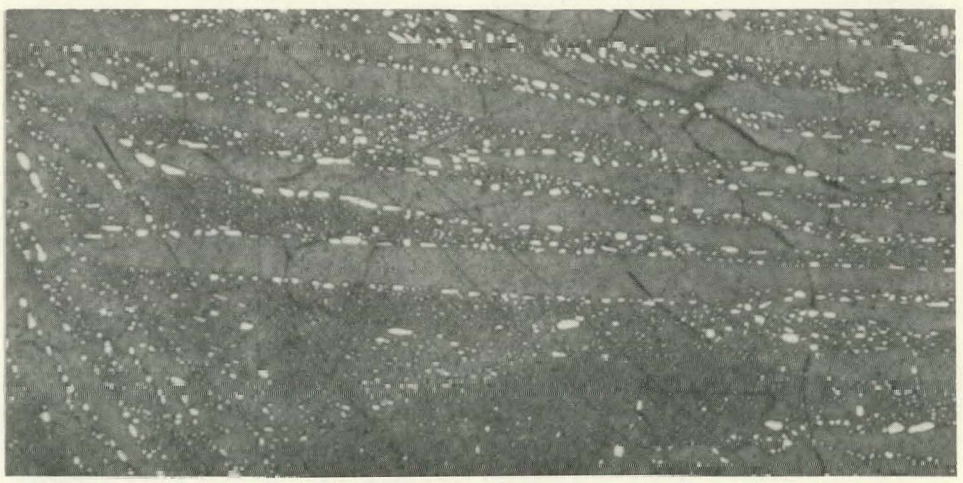
The behavior suggested by Figure 17 (that higher titanium areas may be more sluggish in precipitating U_2Ti) led to an experiment to determine if this relationship might be true. Two small ingots, roughly 20 by 140 by 180 mm, were cast. One ingot contained 0.73 wt % titanium and the other contained 0.95 wt %. Both castings were vacuum homogenized for 24 hours at 1000° C and then rolled at 625° C, with a minimum of reheating, to a thickness of 14 mm. Small specimens of each composition were cut out and wrapped in tantalum foil.



(a) At 675° C. J365-1

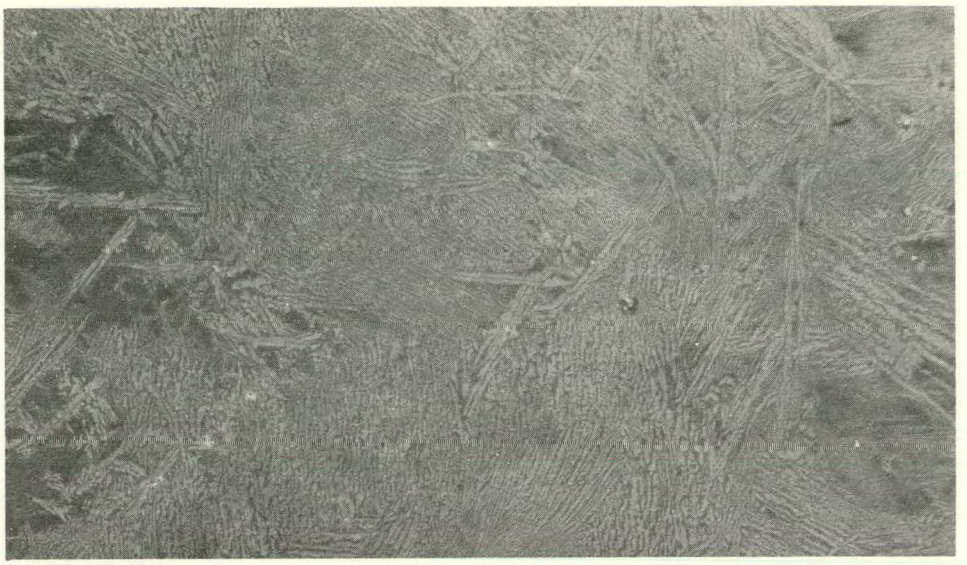


(b) At 695° C. J365-2

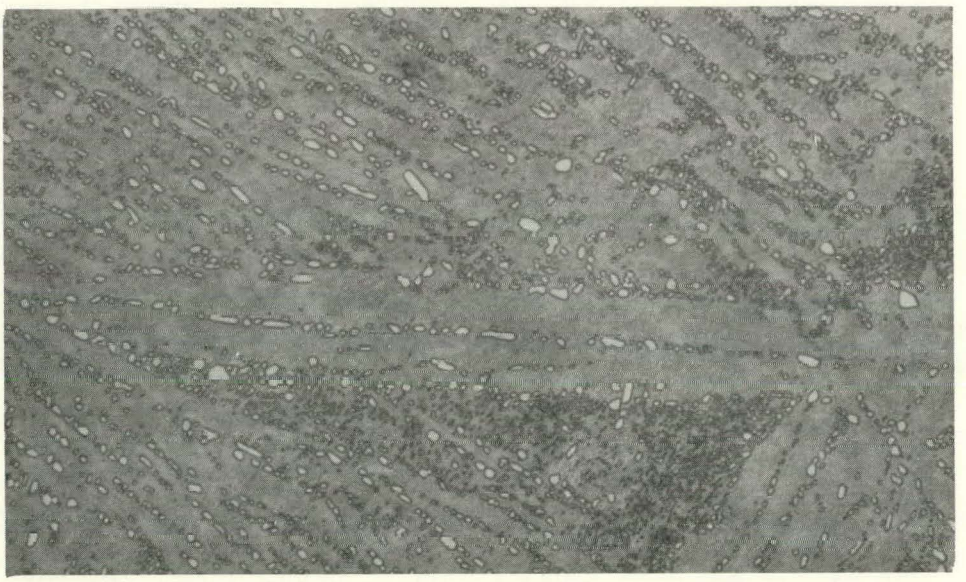


(c) At 715° C. J365-3

Figure 16. CAST URANIUM-0.75 TITANIUM ALLOY THAT WAS TRANSFORMED FOR 72 HOURS IN VACUUM AT THREE TEMPERATURES. (Air Oxidized; Bright Field Illumination; 1000X)



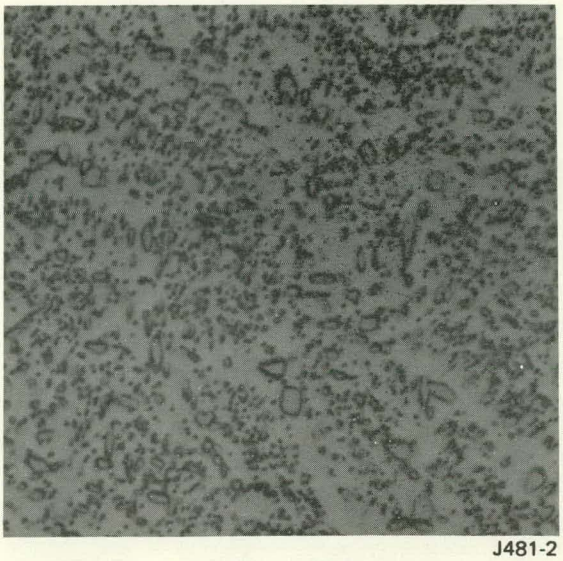
(a) At 100X. J481-1A



(b) At 1000X. J481-1

Figure 17. CAST URANIUM-0.75 TITANIUM ALLOY THAT WAS HEATED AT 695° C FOR 208 HOURS IN VACUUM. (Field of View b is in the Center of View a; Electropolished; Bright Field Illumination)

One specimen of each composition was sealed into a quartz capsule for a transformation heat treatment. Similar specimens were compressively warm worked 50% at 200° C and were also encapsulated. The two capsules were placed adjacent to the control thermocouple in the most uniform area of the furnace and heated for 306 hours at 690 ± 10° C. The microstructures showed no noticeable differences between the hot-rolled and warm-worked structures. The U_2Ti particles were fine and appeared to be smaller and more irresolvable in the U-0.95 Ti alloy.

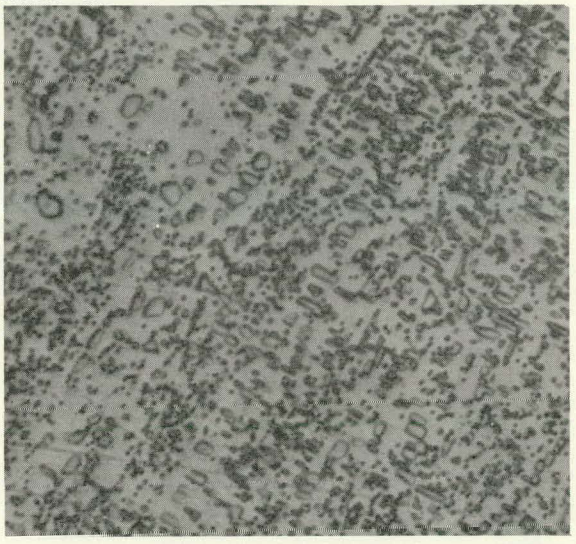


J481-2

(a) Heated for 1 Hr at 800° C in Vacuum, Water Quenched, and Aged 6 Hr at 380° C.

J481-3

(b) Heated 24 Hr at 1000° C in Vacuum.

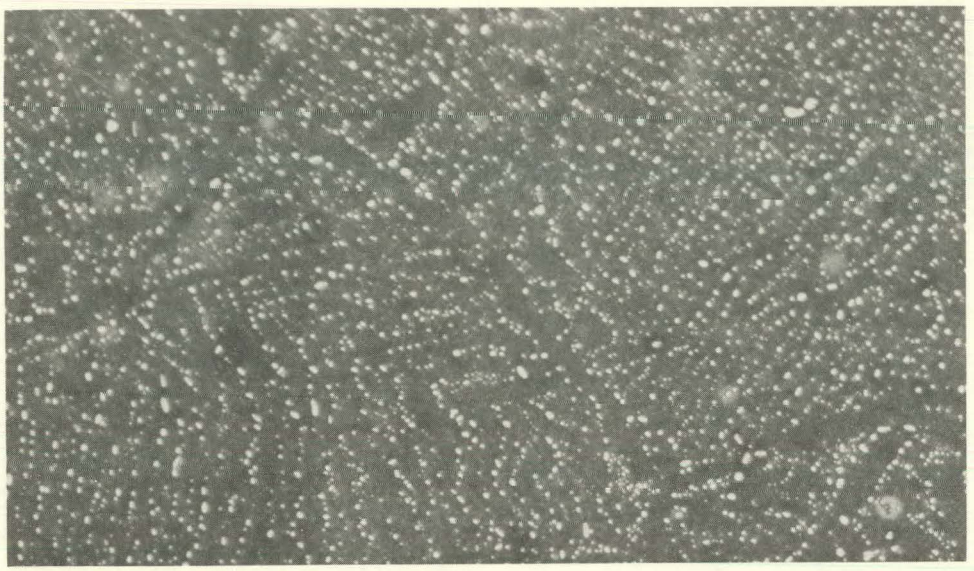


J481-4

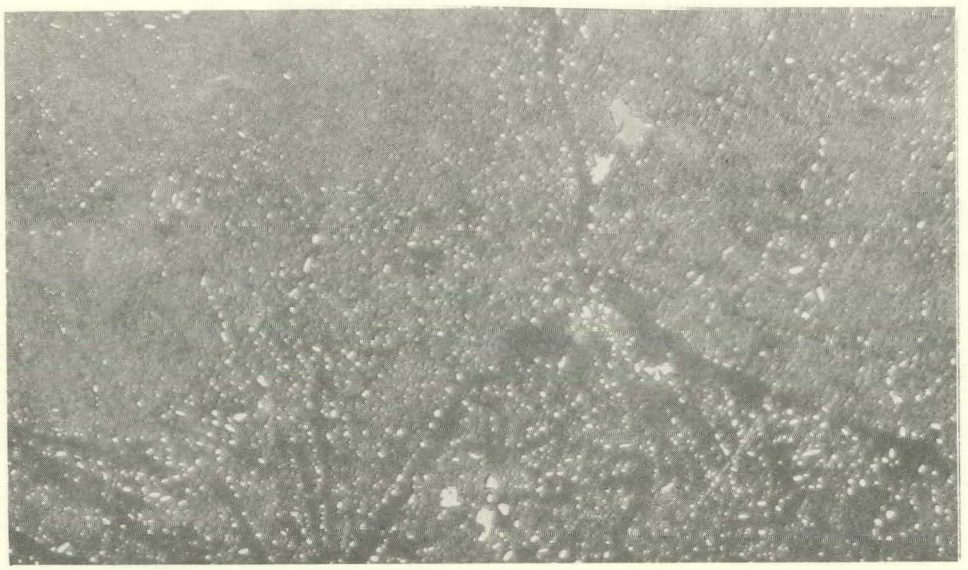
(c) Hot Rolled 55% at 625° C, Heated 24 Hr at 1000° C in Vacuum, Solution Treated 1 Hr at 800° C in Vacuum, Water Quenched, and Aged 6 Hr at 380° C.

Figure 18. CAST URANIUM-0.75 TITANIUM ALLOY THAT WAS TREATED AS DESCRIBED IN THE SUBTITLES AND HEATED IN VACUUM FOR 208 HOURS AT 695 ± 5° C in the (β + Ti) PHASE REGION. (Electropolished; Bright Field Illumination; 1000X)

One further run was made, using the alloys just described, to evaluate the effect of a solution heat treatment in the gamma phase region prior to transformation. Only one capsule containing warm-rolled specimens of each analysis was used in this experiment. The capsule was again placed adjacent to the calibrated control thermocouple and held at 825 ± 10° C for 24 hours and then dropped to 695 ± 10° C for 406 hours. Upon examining the microstructure, the U_2Ti particles in the U-0.73 Ti alloy appeared to be slightly larger and not uniformly spaced. In the U-0.95 Ti alloy there were areas in which the particles appeared to have developed about the same as in the U-0.73 Ti alloy, but more often they were fine and nearly irresolvable. Figure 19 presents photomicrographs of the two microstructures.



(a) U-0.73 Ti Alloy. K255-1



(b) U-0.95 Ti Alloy. K255-2

Figure 19. CAST, HOMOGENIZED, AND ROLLED URANIUM-TITANIUM ALLOYS THAT WERE HEATED IN VACUUM AT 825 ± 10° C FOR 24 HOURS AND AT 695 ± 10° C FOR 406 HOURS. (Air Oxidized; Bright Field Illumination; 500X)

It now appeared that not only were the conditions required to produce large U_2Ti particles very elusive, but also that higher titanium concentrations tended to stabilize the high-temperature gamma phase. Therefore, it was decided to analyze these two specimens with the image analyzer and terminate the experiment. A rectangular area, 6.2 by 6.8 mm, was examined on each sample. This area was evaluated at 900X, using a pattern of 22 scans of 20 butting fields. The scans were separated by 0.04 mm. Each field represented an area of 0.096 mm^2 (at 900X, an area of 255 by 305 mm—very large with

respect to the U_2Ti particle size). Figures 20 and 21 are reproductions of the Quantimet scans. The equivalent of contour lines are drawn in, which show relative locations of the high, medium, and low-titanium-concentration areas on each specimen. The data of Figure 20 suggest that the image-analysis method overestimated the amount of titanium present in the 0.73 wt % Ti alloy; however, those of Figure 21 show much variability and indicate a very much lower titanium concentration than that actually present.

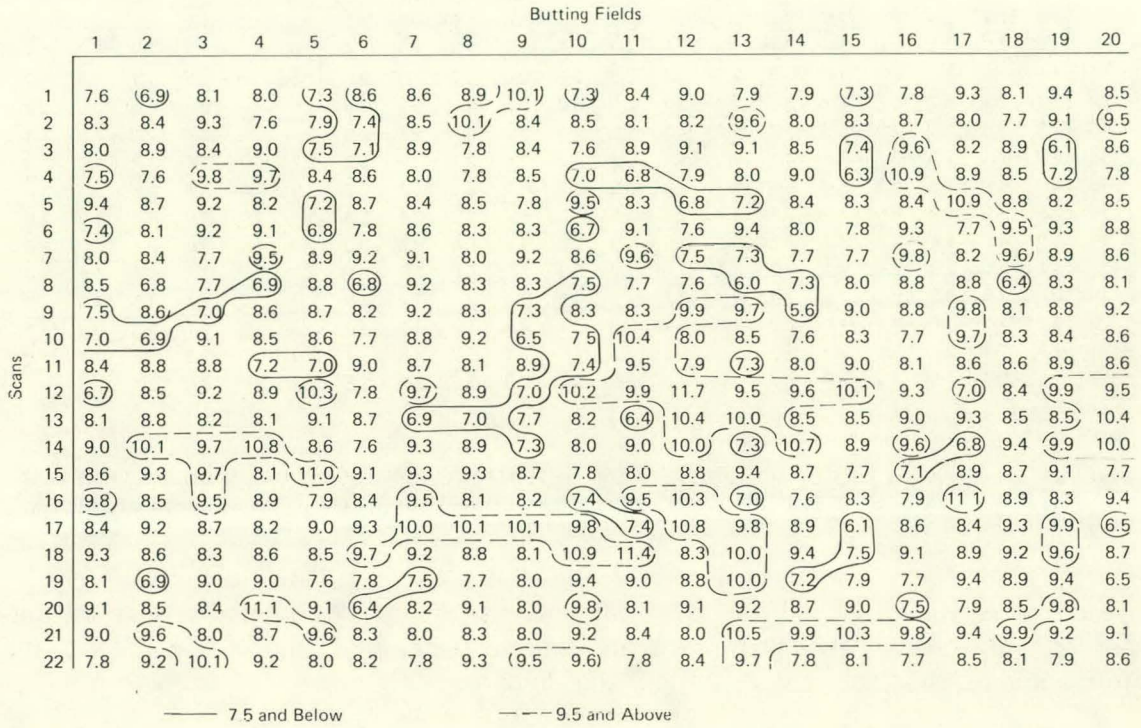


Figure 20. IMAGE ANALYSIS OF VOLUME PERCENT U_2Ti IN URANIUM-0.73 TITANIUM ALLOY THAT WAS HEATED FOR 24 HOURS AT 825 ± 10° C AND FOR 406 HOURS AT 695 ± 10° C. (Overall Average of All Points is 8.6)

Effect of Varying the Heat Treatment on the Homogeneity of the Uranium-0.75 Titanium Alloy

One factor to be assessed in the study of the homogeneity of U-0.75 Ti alloy is the heat treatment needed to promote uniform distribution of the titanium. An experiment was designed to develop this information using mechanical-property data from a cast ingot as a basis for evaluating the heat treatments. The heat-treatment matrix parameters selected were temperatures of 800 and 1000° C for times of 2 and 24 hours.

A 185-mm-diameter, 660-mm-long ingot was prepared by comelting uranium and titanium under vacuum in an induction-melting furnace and casting the melt into a coated graphite mold. After rough machining the side and top, the ingot was sectioned, as illustrated in Figure 22. The nondestructive test (NDT) slices were finish machined with the surfaces flat and parallel, and a chip sample, obtained by a fine machine cut over the entire area

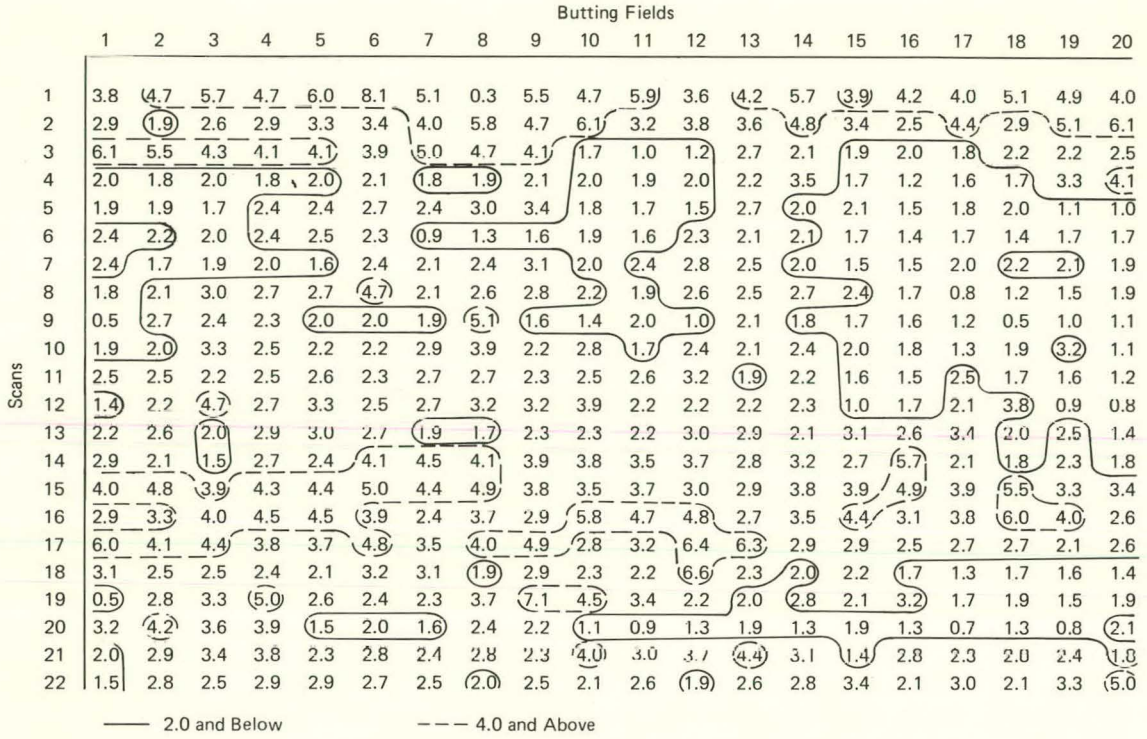


Figure 21. IMAGE ANALYSIS OF VOLUME PERCENT U_2Ti IN URANIUM-0.95 TITANIUM ALLOY THAT WAS HEATED FOR 24 HOURS AT 825 ± 10° C AND FOR 406 HOURS AT 695 ± 10° C. (Overall Average of All Points is 2.8)

of each slice, was collected for chemical analysis. At the top, middle, and bottom locations, respectively, the titanium analyses were 0.82, 0.83, and 0.81 wt % and the corresponding carbon values were 24, 30, and 38 ppm.

Radiographs of the three slices revealed no porosity or cracks. The coarse-grained character of the ingot was evident at all three locations, and there appeared to be an anomalously low-density area in the center of the slices which was more marked at the bottom and center than at the top. To check the possibility that thickness differences might account for the variability, chemistry and density specimens were taken from the edge and center of each slice. The results appear in Table 3 and reveal that the titanium concentration actually was lower and the density higher at the center of the ingot, with no top-to-bottom variability.

Table 3
CHEMISTRY AND DENSITY DATA FROM THE EDGE AND CENTER OF NONDESTRUCTIVE TEST SLICES FROM A URANIUM-0.75 TITANIUM ALLOY INGOT

Sample Location	Density (g/cm^3)	Titanium Content (wt %)	Carbon Content (ppm)
Top Center	18.61	0.76	32
Top Edge	18.43	0.79	50
Middle Center	18.65	0.76	21
Middle Edge	18.60	0.79	32
Bottom Center	18.85	0.76	48
Bottom Edge	18.54	0.80	56

The four combinations of time/temperature conditions were all to be tested on bars from the top billet (Figure 22) of the cast ingot. Both the top and middle billets were sliced into tensile-bar blanks, as

pictured in Figure 23, and a four-character identification was applied to each blank. The first letter designated the location of the blank in the ingot, the second letter indicated the quadrant, while the two numbers specified the location of the tensile-bar blank within the quadrant. Eight blanks in each quadrant were tested. Blanks 11, 12, 21, and 22 represented the interior of the ingot; Blanks 15, 25, 51, and 52, the exterior. Table 4 summarizes the experimental conditions and the test-specimen source. The tensile-test results are listed in Table 5. Except for ductility, superior properties were produced in the unhomogenized alloy; however, the best combination of properties was shown by specimens which had been subjected to the 1000° C 24-hour homogenization treatment.

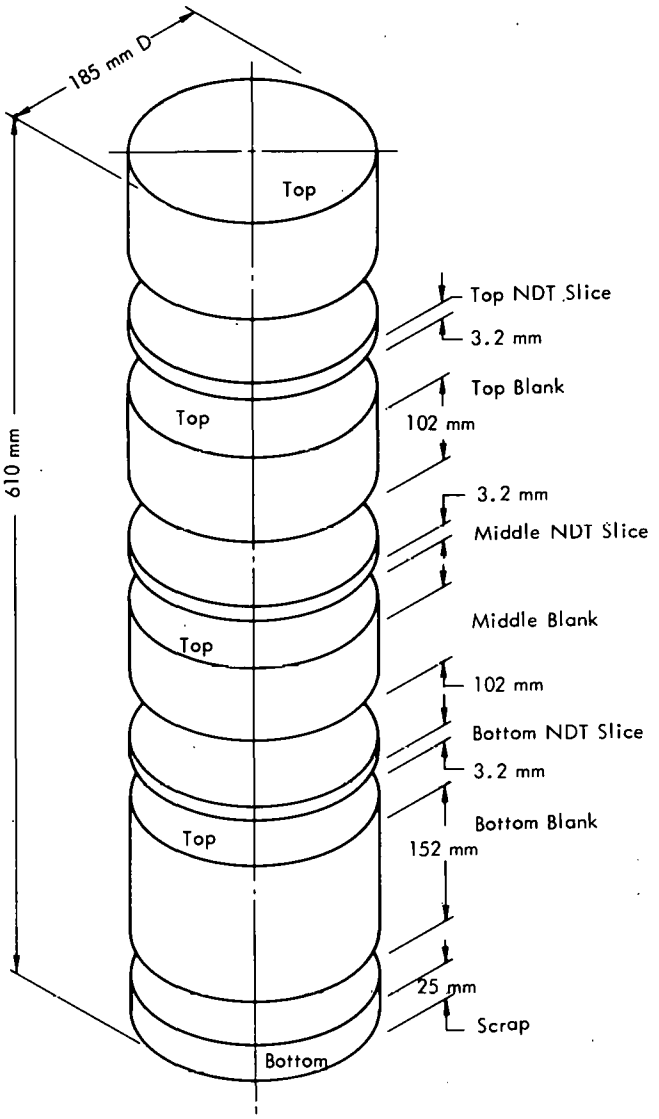


Figure 22. NONDESTRUCTIVE TESTING SLICE AND BLANK LOCATIONS IN AN INDUCTION-CAST URANIUM-0.75 TITANIUM ALLOY INGOT.

An analysis of variance was run on all the tensile date in Table 5 (including the as-cast data) to determine whether there were significant differences in properties as a function of the homogenization treatment and location of test specimens within the ingot. Statistically significant differences were observed for all properties as a function of the homogenization treatment. Significant differences as a function of location were noted only for the 0.2% offset and 0.85% extension yield strengths. For the specimens located in the interior of the ingot, the average yield strengths were 809 and 846 MPa; corresponding values for the specimens near the outer surface were 838 and 870 MPa.

An attempt was made to optimize the holding time for the 1000° C homogenization treatment by heat treating two specimens from Location TC (Figure 23) for each of the times: 3, 7, 16, 30, and 70 hours. The resulting data revealed little difference among all the treatments and were inconclusive, but there appears that there is little or nothing to be gained by extending the times beyond about 16 hours. The tensile specimens for the heat-treatment study were 6.4 mm (0.252 in) in gage diameter and 25.4 mm (1 in) in gage

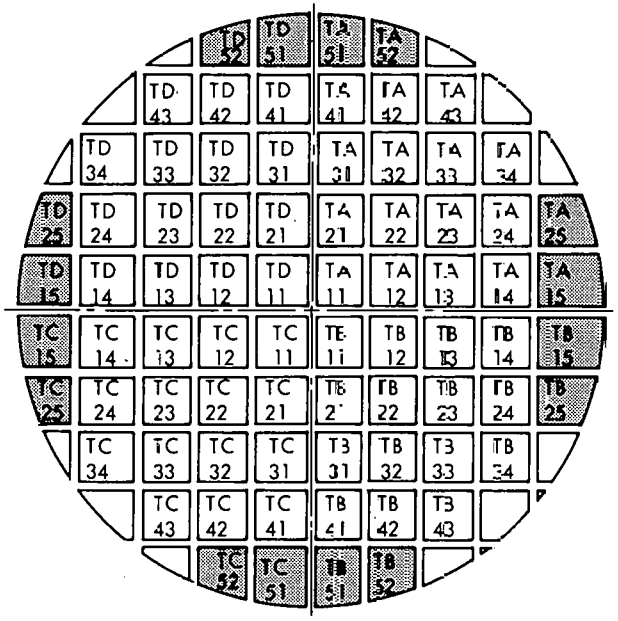


Figure 23. MANNER IN WHICH BILLETS FROM THE URANIUM-0.75 TITANIUM ALLOY INGOT WERE CUT INTO TENSILE-BAR BLANKS. (This Figure Represents the Top Blank; for the Middle Billet, an "M" was Used in Place of the "T")

Table 4

HEAT TREATMENTS AND SPECIMEN MATERIAL SOURCE FOR THE URANIUM-0.75 TITANIUM ALLOY HOMOGENIZATION STUDY

Heat Treatment	Blank and Quadrant	Tensile-Bar Blank Numbers
800° C for 2 Hr in Vacuum; 800° C for 1 Hr in Vacuum Water Quench; 380° C for 6 Hr in Vacuum	Top Blank, Quadrant A (TA)	TA11, TA12, TA21, TA22 TA15, TA25, TA51, TA52
1000° C for 24 Hr in Vacuum; 800° C for 1 Hr in Vacuum, Water Quench; 380° C for 6 Hr in Vacuum	Top Blank, Quadrant B (TB)	TB11, TB12, TB21, TB22 TB15, TB25, TB51, TB52
800° C for 24 Hr in Vacuum; 800° C 1 Hr in Vacuum, Water Quench; 380° C for 6 Hr in Vacuum	Top Blank, Quadrant C (TC)	TC11, TC12, TC21, TC22 TC15, TC25, TC51, TC52
1000° C for 2 Hr in Vacuum; 800° C for 1 Hr in Vacuum, Water Quench; 380° C for 6 Hr in Vacuum	Top Blank, Quadrant D (TD)	TD11, TD12, TD21, TD22 TD15, TD25, TD51, TD52
800° C for 1 Hr in Vacuum, Water Quench; 380° C for 6 Hr in Vacuum	Middle Blank, Quadrant B (MB)	MB11, MB12, MB21, MB22 MB15, MB25, MB51, MB52
As Cast	Middle Blank, Quadrant A (MA)	MA11, MA12, MA21, MA22 MA15, MA25, MA51, MA52
1000° C for 24 Hr in Vacuum; 800° C for 1 Hr in Vacuum, Water Quench; 380° C for 6 Hr in Vacuum	Bottom Blank (B)	B1, B2, B3, B4, B5, B6, B7, B9 B10, B11, B12, B13, B16, B17, B18

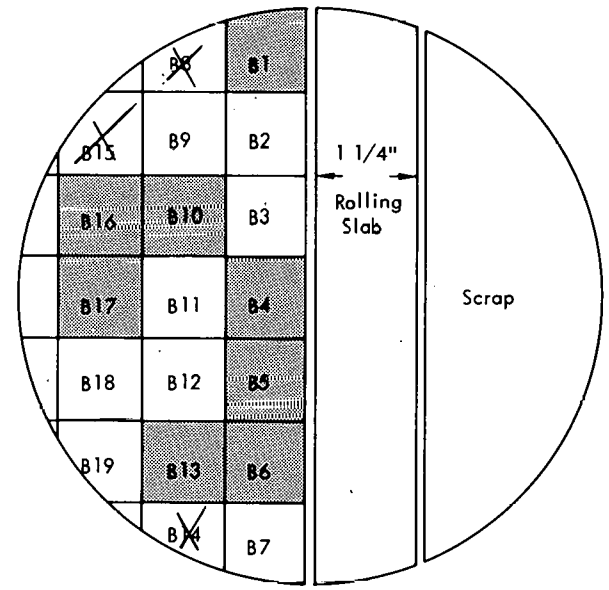
Table 5

TENSILE PROPERTIES OF CAST URANIUM-0.75 TITANIUM ALLOY AFTER HOMOGENIZATION

Homogenization Treatment[1]	Specimen Identification	Ultimate Tensile Strength (MPa)	Yield Strength[2] (MPa)	Yield Strength[3] (MPa)	Elongation[4] (%)	Reduction in Area (%)
2 Hr - 800° C	TA11	1311	767	822	12.0	9.3
	TA12	1246	784	842	7.0	9.7
	TA21	1240	759	821	8.0	8.6
	TA22	1310	762	825	13.0	11.2
	TA51	1279	816	867	8.0	8.3
	TA52	1344	802	860	9.0	6.7
	TA25	1213	799	843	7.0	7.0
	TA15	1273	858	896	6.0	7.7
	Avg	1277	794	847	8.8	8.6
	95% CL	±37	±28	±22	±2.1	±1.2
24 Hr - 1000° C	TB11	1361	816	851	18.5	25.8
	TB12	1388	836	879	12.0	12.3
	TB21	1399	890	918	16.0	20.2
	TB22	1421	879	908	16.5	13.4
	TB51	1428	890	929	19.5	17.4
	TB52	1427	890	917	15.0	15.2
	TB25	1406	863	903	19.0	24.4
	TB15	1449	896	922	17.0	15.2
	Avg	1410	871	903	16.7	18.0
	95% CL	±23	±25	±21	±2.0	±4.2
24 Hr - 800° C	TC11	1366	832	865	9.5	10.7
	TC12	1340	883	905	8.5	10.0
	TC21	1342	846	874	10.0	10.4
	TC22	1375	898	918	7.5	8.3
	TC51	1324	895	917	5.5	7.0
	TC52	1412	932	939	9.5	7.0
	TC25	1339	864	889	10.0	9.0
	TC15	1366	918	939	6.5	7.0
	Avg	1358	884	906	8.4	8.7
	95% CL	±23	±30	±23	±1.4	±1.3
2 Hr - 1000° C	TD11	1395	887	905	13.0	12.3
	TD12	1427	884	905	17.0	20.2
	TD21	1406	917	929	11.0	11.5
	TD22	1453	905	920	15.0	15.9
	TD51	1448	936	943	12.0	12.7
	TD52	1464	945	949	13.5	13.0
	TD25	1394	896	905	13.0	12.2
	TD15	1445	918	932	12.0	11.5
	Avg	1429	911	923	13.3	13.7
	95% CL	±23	±19	±15	±1.6	±2.5
None	MB11	1391	969	969	4.0	4.7
	MB12	1459	1011	1014	4.0	5.1
	MB21	1395	905	946	8.0	5.4
	MB22	1431	970	991	5.0	5.1
	MB51	1461	995	995	6.0	5.8
	MB52	1424	1035	1022	3.0	2.7
	MB25		Defect in Specimen			
	MB15	1386	981	998	3.0	3.5
	Avg	1421	981	991	4.7	4.6
	95% CL	±29	±36	±24	±1.7	±1.0
As Cast	MA11	763	484	559	3.3	1.6
	MA12	794	502	578	2.5	2.0
	MA21	808	508	579	2.5	2.4
	MA22	790	513	591	3.0	2.4
	MA51	818	497	580	3.0	1.6
	MA52	783	491	574	3.0	1.2
	MA25	818	493	580	3.0	1.6
	MA15	812	492	573	2.5	1.6
	Avg	799	497	577	2.8	1.8
	95% CL	±19	±10	±9	±0.3	±0.4

(1) All specimens except "as cast" were heated at 800° C for 1 hr, water quenched, then aged at 380° C for 6 hr to develop properties.
(2) At 0.2% offset.
(3) At 0.85% extension.
(4) In a 25.4-mm gage length.

length. Because of the coarse grain size of the cast ingot, it was felt that there might be a disproportionate effect of grain size on the tensile-test results. An attempt to investigate this possibility was made by preparing both 6.4 and 12.7-mm-gage-diameter specimens from the bottom billet. The locations of these specimens are indicated in Figure 24, and the heat treatment is shown in Table 4. Table 6 contains the tensile data. The small-gage-diameter specimens were superior to the large specimens, particularly with respect to elongation and reduction in area; but, for the small specimens, the confidence limits on the mean were greater for all properties except tensile strength.



Note:

Specimens B2, B3, B7, B9, B11, B12, B18, and B19 were 6.4 mm in Gage Diameter;

Specimens B1, B4, B5, B6, B10, B13, B16, and B17 were 12.8 mm in Gage Diameter;

Specimens B8, B14, and B15 were not used.

Figure 24. MANNER IN WHICH THE BOTTOM BILLET FROM THE URANIUM-0.75 TITANIUM INGOT WAS CUT INTO TENSILE-BAR BLANKS AND A ROLLING SLAB. (Specimens B2, B3, B7, B9, B11, B12, B18, and B19 were 6.4 mm in Gage Diameter; Specimens B1, B4, B5, B6, B10, B13, B16, and B17 were 12.8 mm in Gage Diameter; Specimens B8, B14, and B15 were not Used)

The rolling slab from the bottom blank (Figure 24) was hot rolled in the high-alpha temperature range in the direction perpendicular to the cylindrical axis of the ingot. The final thickness was 14.0 mm. Three bars from an end of the plate and four from the center were cut in a direction parallel to the original ingot axis in order to represent the surface and center of the ingot.

The purpose of running tests on wrought material was simply to compare the properties of cast and wrought alloy.

The tensile-bar blanks were homogenized at 1000° C for 24 hours in vacuum and given the standard heat treatment of one hour in vacuum at 800° C and water quenched, then

Table 6
TENSILE PROPERTIES OF CAST, HOMOGENIZED, AND HEAT-TREATED URANIUM-0.75 TITANIUM ALLOY AS DETERMINED USING THE TWO SIZES OF TENSILE BARS

Specimen Identification	Gage Diameter (mm)	Ultimate Tensile Strength (MPa)	Yield Strength[1] (MPa)	Yield Strength[2] (MPa)	Elongation[3] (%)	Reduction in Area (%)	Remarks
B1	12.8	1362	929	938	5.0	6.6	
B4		-	-	-	-	-	Defective Specimen
B5		1422	901	925	12.0	13.0	
B6		1424	921	960	11.0	11.1	(4)
B10		1357	839	865	12.5	13.7	(4)
B13		1282	832	884	5.5	6.8	(4)
B16		1230	891	916	2.5	3.1	(4)
B17		1307	860	881	5.5	6.6	(4)
Mean		1341	882	945	7.7	8.7	
95% CL		±67	±36	±32	±3.7	±3.6	
B2	6.4	1432	943	971	9.5	9.3	
B3		-	-	-	-	-	Defective Specimen
B7		-	-	-	-	-	Defective Specimen
B9		1466	936	954	17.5	17.1	
B11							
B12		1402	886	896	18.5	18.5	Defective Specimen
B18		1415	902	909	16.0	17.4	
Mean		1430	917	932	15.4	15.6	
95% CL		±43	±43	±57	±6.5	±6.6	

(1) At 0.2% offset.
(2) At 0.85% extension.
(3) In a 25.4-mm gage length.
(4) A small spherical void (< 0.5 mm D) was near the center of the fracture surface.

380° C in vacuum for six hours. Tensile-test results are given in Table 7. The wrought specimens have properties which are apparently no different from the as-cast alloy which received the 1000° C, 24-hour homogenization treatment.

Because the homogenization treatment had been given subsequent to the hot working, the resultant structure was probably very nearly as coarse grained as it had been as cast. To more definitely examine the effect of working, a homogenization treatment prior to working would be more appropriate. Material for such an experiment was prepared by using some of the nominally U-0.75 Ti alloy slab which had been cast, homogenized, and rolled for the earlier heat-treatment portion of this study. As mentioned previously, the analysis showed the titanium level to be 0.73 wt % and the carbon content, 40 ppm. As stated earlier, the ingot was vacuum homogenized at 1000° C for 24 hours and rolled out of a 625° C salt bath, with minimum reheating, to 14-mm-thick plate. Five 13-mm-diameter bars were machined from the rolled plate, heated at 800° C for one hour in vacuum, water quenched, and aged at 380° C for six hours in vacuum. Tensile bars (6.4-mm gage diameter) were machined and tested, with the results given in Table 8. The strengths were lower, but the ductility higher than for the cast alloy. The scatter among individual tests is also considerably less. Compared with the as-cast grain size, the grain size of the material was small, as noted in Figure 25.

Table 7

TENSILE PROPERTIES OF WROUGHT, THEN HOMOGENIZED URANIUM-0.75 TITANIUM ALLOY

Specimen Identification	Location in Ingot	Ultimate Tensile Strength (MPa)	Yield Strength[1] (MPa)	Yield Strength[2] (MPa)	Elongation[3] (%)	Reduction in Area (%)
E1	Surface	1393	870	895	17.0	20.7
E2	Surface	1383	847	890	16.0	23.7
E3	Surface	1420	885	921	16.0	17.7
Mean		1399	867	902	16.3	20.7
95% CL		±13	±47	±42	±1.4	±2.1
M1	Center	1392	870	914	12.5	10.5
M2	Center	1409	871	906	15.5	14.3
M3	Center	1390	857	892	16.0	17.9
M4	Center	1381	836	874	15.0	15.2
Mean		1393	858	896	14.8	14.5
95% CL		±18	±26	±28	±2.5	±4.9

(1) At 0.2% offset.
(2) At 0.85% extension.
(3) In a 25.4-mm gage length.

Table 8

TENSILE PROPERTIES OF HOMOGENIZED, THEN WROUGHT URANIUM-0.75 TITANIUM ALLOY

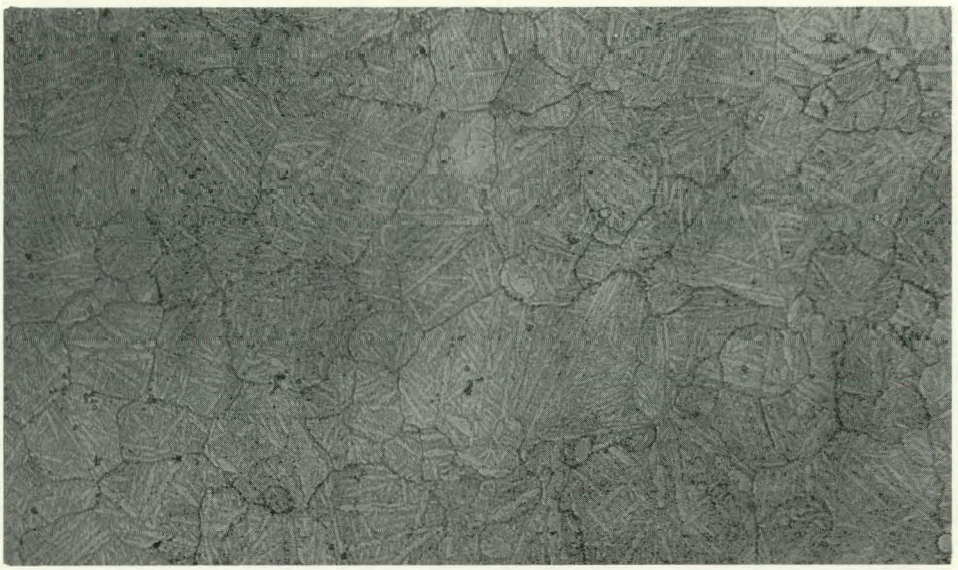
Specimen Identification	Ultimate Tensile Strength (MPa)	Yield Strength[1] (MPa)	Elongation[2] (%)	Reduction in Area (%)
1721	1370	774	25.0	39.6
1722	1343	785	25.5	42.4
1723	1359	780	27.0	41.2
1724	1351	782	26.0	40.8
1725	1357	796	24.0	39.6
Mean	1356	703	25.3	40.7
95% CL	±12	±10	±1.4	±1.5

(1) At 0.2% offset.
(2) At 0.85% extension.

DISCUSSION OF RESULTS

Microsegregation Analysis

The coring or microsegregation revealed by the EB microprobe in both the as-cast U-0.5 Ti and U-0.75 Ti alloys was readily leveled by the 800° C, one-hour solution treatment and was not further affected by the aging treatment used to strengthen the alloy. The longer and higher-temperature homogenization treatment (1000° C, 24 hours) appears to have further reduced the extent of the high and low-titanium areas in the U-0.75 Ti alloy. The decrease was small, presumably because once the initially high, local



K455-1A

Figure 25. URANIUM-0.73 TITANIUM ALLOY THAT WAS HOMOGENIZED IN VACUUM AT 1000° C FOR 24 HOURS, HOT AND WARM ROLLED TO 30% REDUCTION, SOLUTION TREATED IN VACUUM FOR ONE HOUR AT 800° C, AND AGED FOR SIX HOURS AT 380° C. (Etched; Bright Field Illumination; 100X)

concentration gradients were reduced, the driving force for diffusion was greatly diminished and the process proceeded at a continually decreasing rate.

There were several findings which revealed that longer-range variations (over distances on the order of millimeters) existed in the level of titanium within the alloy ingot, even after homogenization. Primary among these was the approximately 0.1 wt % titanium difference between the adjacent specimens from which the data of Figures 6 and 7 were obtained. Lesser variations, over shorter distances, appear within the specimen, which was the source of the data in Figure 5. When the analyzed areas of the specimens used for Figures 6 and 7 were greatly extended to produce the data in Figures 10 and 9, respectively, a few areas were found in the specimen of Figures 6 and 10 which were as low in titanium as the specimen of Figures 7 and 9 (ie, in the range of 0.7 wt % titanium), but no high-titanium areas were detected in the specimen of Figures 7 and 9.

Thermal homogenization treatments which would level the compositional differences existing over wavelengths on the order of millimeters would be completely unreasonable from the time involved. The solution to this problem is the development of casting procedures which assure complete solution of the titanium and homogeneity of the melt prior to casting.

Attempt to Develop a Method for Quantitatively Measuring Long-Range Titanium Segregation

The method by which an equilibrium structure of a consistently resolvable U_2Ti precipitate in alpha uranium solid solution could be obtained was not fully developed. The benefit to be derived was not worth a continued effort, especially since concurrent

development of casting methods was resulting in improved alloy homogeneity. The method is felt to have some potential, however, because, assuming that the structure of Figure 12 closely approached an equilibrium structure, it is ideal for a Quantimet analysis since the white U_2Ti particles are large and resolvable and a definite color difference can be developed between the two phases.

Using Figure 13, the titanium data in Table 1 is seen to vary substantially from 0.70 to 1.22 wt % within the area analyzed. The average of 0.86 wt % is high, compared with the desired analysis. Irregular cyclic trends in titanium concentration occur in the data in Table 2 for the 4-mm-long bands analyzed. As seen in Figure 14, the bands did not show a monotonic trend in the data, so the cycles are less than 4 mm in wavelength and appear to be on the order of 1 to 2 mm. Of additional interest is the fact that the averages of the data in Table 2 (using Figure 13) show a titanium content of about 0.79 wt %. The difference between the averages of the data of Tables 1 and 2, from adjacent locations on the same specimen, is on the same order as the difference between the adjacent specimens from which the data of Figures 6 and 7 were obtained. This result is a further indication that variations on the order of 0.1 wt % titanium may exist over ranges on the order of millimeters or tens of millimeters within a cast ingot.

Incomplete transformation of the 1000° C, 24-hour homogenized specimen of Figure 15 and the complete lack of the desired structure in the accompanying specimen which had been water quenched after one hour at 800° C apparently was poor temperature control such that the temperature rose above the eutectoid temperature. Though of nearly eutectoid composition, the Figure 15 specimen shows the behavior of a hypoeutectoid alloy. Similar behavior has been reported as being caused both by the cooling rate[1] and by impurities;[2] ie, these factors apparently act to displace the eutectoid composition to the right. Since the dark, untransformed phase was analyzed to contain 1.27 wt % titanium, this is probably close to the effective eutectoid composition. Assuming that the light, beta-phase islands (which contain 0.29 wt % titanium) constitute about 50 vol % of the light-dark, two-phase area, the average composition would be about 0.75 wt % titanium—close to the alloy analysis. With the eutectoid reported as being at 0.7[1] to 0.8 wt %[2] titanium, no explanation can be offered for the hypoeutectoid behavior of the alloy.

Similar hypoeutectoid characteristics were observed in all as-cast samples used in the study; but, in some of the samples pretreated at 800 or 1000° C, the U_2Ti dispersion was rather uniform; eg, the three views of Figure 18 and View a of Figure 19, which are more indicative of a eutectoid decomposition. In all but the specimens of Figures 12 and 15 there were areas of fine U_2Ti. The specimen of Figure 15, being accidentally heated to the eutectoid temperature, probably very slowly passed through or was cycled up and down through the temperature. Although it is not suspected that this happened with the specimen of Figure 12, such an occurrence might account for the large U_2Ti particle size.

Figures 19 through 21 are the photomicrographs and Quantiment data from the 0.73 and 0.95 wt % titanium alloys. These figures indicate another shortcoming of attempting to quantitatively analyze for titanium at a given location by measuring the volume of U_2Ti present. The higher titanium alloy shows less, or at least less resolvable, U_2Ti in the microstructures, so there is an apparent tendency for higher titanium concentrations to

stabilize the gamma phase so that it resists decomposition even after very long times at temperature. This fact is also reflected in the Figure 21 Quantimet data. Note, too, in View b of Figure 19 that the U-0.95 Ti alloy, in spite of its being hypoeutectoid, according to a published phase diagram,(2) shows grain boundaries free of U_2Ti. This hypoeutectoid behavior is contrary to the expected decomposition structure; but, as mentioned earlier, is not contrary to some reported experimentation.

Effect of Varying the Heat Treatment on the Homogeneity of the Uranium-0.75 Titanium Alloy

The apparent low-density areas observed by radiography in the centers of the NDT slices must have been due to a thinned area, which, in itself, might have been a consequence of a change in machining characteristics imparted to the alloy by the lowered titanium concentration. The radial titanium gradients were expected, but the anticipated top-to-bottom, low-to-high titanium gradient was not observed.

In spite of the fact that short annealing times at 800° C have been shown to eliminate microsegregation or coring, the 1000° C, 24-hour treatment was much more effective in bringing about improvement in properties. These findings would tend to suggest that longer-wavelength macrosegregation exists and suggests that it is this type of segregation which most influences the mechanical properties and requires higher temperatures and longer times to smooth out. The failure of very long times at 1000° C to further improve the elongation and reduction in area of cast alloy indicates that, beyond a certain point, the large grain size and anisotropy of the orthorhombic crystal structure became controlling factors. This consequence would be particularly true when the average grain diameter approaches a substantial fraction of the tensile-bar diameter. That a grain-size/test-bar-diameter relationship existed appears to have been borne out by the property determination using two sizes of test bars. The lower-ductility data from the larger-diameter bars were due to a size-effect difference between the two sizes of tensile bars, but the decrease in variability in the data from the large bars was probably a consequence of a greater number of grains in the cross section of the bar.

The effect of grain size on properties is at least partially indicated by the properties of the cast and rolled slab in Table 8. A comparison, however, is complicated by the small size of the casting used since segregation would be expected to be less severe in a thin slab than in a large, cylindrical ingot. The principal effect of grain size appears to be in the ductility and reduction-in-area properties. Yield strength, for the heat treatment employed, is apparently more directly related to titanium content, as revealed by the statistical analysis of specimens from the inside and outside of the ingot. The size of the alloy bars when heat treated, in particular when water quenched, is also probably an important variable in the property/titanium-content relationship. The large bars from the bottom section of the ingot (Figure 24 and Table 6) show higher yield strengths for the same average titanium content than did the smaller bars from the top and middle sections.

CONCLUSIONS

The following conclusions can be enumerated as a result of this study:

1. A homogenization treatment of 1000° C for 24 hours given to as-cast alloy was superior to 800° C for 2 and 24 hours, and 1000° C for 2 hours, as determined by tensile properties.

2. Extending the holding time at 1000° C to 70 hours was ineffective in improving the properties of the alloy, possibly because the large grain size limited the amount of improvement possible.

3. Microsegregation due to coring (nonequilibrium solidification) may be readily smoothed by heat treatments of 800° C for 1 hour for both the 0.5 and 0.75 wt % titanium alloys. However, the 1000° C, 24-hour treatment is required to effectually further smooth the chemical inhomogeneities.

4. Long-range, cyclic variations on the order of 0.1 wt % titanium may exist over wavelengths on the order of millimeters to tens of millimeters, even after a 1000° C, 24-hour homogenization. Reasonable homogenization treatments are ineffective in eliminating this type of variability.

5. All tensile properties are affected by grain size and the tensile-bar dimensions. Yield strength appears to be most sensitive to small titanium variations.

6. Holding U-0.75 Ti alloy in the ($\beta + U_2Ti$) phase field for times of 72 to 406 hours and water quenching produced a structure of variable-size U_2Ti particles in a uranium/titanium solid solution matrix. An EB microprobe analysis of the solid solution showed the titanium concentration to be 0.29 ± 0.06 wt %, close to the published literature value for the solubility of titanium in a beta solid solution.[1]

7. All of the factors controlling U_2Ti precipitation and particle size were not determined, so that large, resolvable particles for quantitative metallographic analysis could not be consistently attained. Titanium content appeared to be one controlling factor, with higher concentrations tending to retard the $\gamma \rightarrow \beta + U_2Ti$ transformation and result in smaller, less resolvable U_2Ti particles.

8. When a resolvable U_2Ti particle structure was attained, a quantitative metallographic analysis indicated a greater concentration of titanium (assuming 0.3 wt % in solution and U_2Ti stoichiometry) than was obtained by chemical analyses.

9. Although U-0.75 Ti alloy is reportedly of eutectoid composition, many of the metallographic structures obtained showed hypoeutectoid behavior. This difference in result was more apt to happen when an as-cast structure rather than a 1000° C, 24-hour homogenized structure was transformed in the ($\beta + U_2Ti$) phase field.

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