

27/18
1/3/78
2/5 ug. to NTS
MASTER

Y/DA-7421

**A STUDY OF VARIABLE ELONGATIONS
IN THE URANIUM-0.75 WEIGHT
PERCENT TITANIUM ALLOY**

T. G. Kollie

November 1977



**OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE**

*prepared for the U.S. DEPARTMENT OF ENERGY under
U.S. GOVERNMENT Contract W-7405 eng 26*

DISCLAIMER

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

DISCLAIMER

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

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Date of Issue: November 28, 1977

Y/DA-7421

**A STUDY OF VARIABLE ELONGATIONS
IN THE URANIUM-0.75 WEIGHT
PERCENT TITANIUM ALLOY**

T. G. Kollie

**Metallurgy Department
Y-12 Development Division**

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

**Oak Ridge Y-12 Plant
P. O. Box Y, Oak Ridge, Tennessee 37830**

**Prepared for the US Energy Research
and Development Administration
Under US Government Contract W-7405-eng-26**

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

EB

CONTENTS

SUMMARY	3
INTRODUCTION	4
A STUDY OF VARIABLE ELONGATIONS IN A URANIUM-TITANIUM ALLOY	5
Specimen Fabrication	5
Results	6
Discussion	10
Conclusions and Recommendations	13
REFERENCES	14

SUMMARY

Tensile specimens cut from the same gamma-quenched-and-aged, 32-mm-thick plate of U-0.75 wt % Ti (U-0.75 Ti) alloy exhibited elongations that varied between 9.4 and 17.2%. Results of microhardness, electron microprobe, chemical analysis, scanning electron microscopy, transmission electron microscopy, ion microprobe analysis, optical microscopy, heat-transfer calculations, and quenching-rate studies led to the conclusion that the variable ductilities were caused by quenching rates from the gamma phase that were too slow and were different across the thickness of the plate. Work of others is cited to support this conclusion. Their work showed that quenching rates of 8 to 50° C/s produced a duplex microstructure of α' and $\alpha + U_2Ti$ phases in the U-0.75 Ti alloy, with ductilities between 7 and 26%, respectively. Two solutions to the problem are suggested: (1) add a third element to the alloy to slowdown the $\gamma \rightarrow \alpha + U_2Ti$ transformation so that titanium is retained in solution in the martensitic α' phase, or (2) substantially improve the heat-transfer coefficients in the quenching bath. The latter suggestion is the near-term solution, whereas the former suggestion is the long-range and better solution.

INTRODUCTION

Uranium-0.75 weight percent titanium alloy is an age-hardenable alloy having high strength and density. A two-step heat treatment is used to optimize the mechanical properties of this alloy. In the first step, the alloy is quenched from the gamma-stable phase field ($\sim 800^\circ \text{ C}$) in water; the second step is to age the alloy in the α - γ_2 two-phase region (~ 300 to 500° C). If the quenching rate from the gamma-phase region is below about 200° C/s , titanium precipitates from the solid solution as the compound U_2Ti , resulting in a loss in aging response, strength, and elongation.¹⁻³

Elongation of the U-0.75 Ti alloy is particularly sensitive to the quenching rate.^{1,2} For example, a quenching rate of 50° C/s retains 75% by volume of the alloy, with titanium in solution in a martensitic (α') structure, for which the aged alloy has an elongation of 25%. As the quenching rate falls to 5° C/s , little titanium is retained in solution and the elongation of the aged alloy is decreased to 7%.

Recently, four tensile specimens from a 32-mm-thick plate of the U-0.75 Ti alloy exhibited elongations that varied between 9.4 and 17.2%. Results presented in this report show that these variable elongations were caused by quenching rates from the gamma-phase region that: (1) were too slow, and (2) varied across the thickness of the plate.

A STUDY OF VARIABLE ELONGATIONS IN A URANIUM-TITANIUM ALLOY

SPECIMEN FABRICATION^(a)

The U-0.75 Ti alloy was cast into 89 x 140 x 64-mm brick ingots which were homogenized at 1000° C for four hours. The ingots were rolled into 203 x 114 x 32 mm plates which were solution annealed at 800° C for six hours, then quenched in water from 800° C (gamma quenched).

Seven 32-mm-diameter cylinders were cut from the plate, with the longitudinal axis of the cylinder parallel to the largest dimension of the plate. A 2% upset was applied to the cylinders for "stress leveling", and the cylinders were aged at 370° C for eight hours. As illustrated in Figure 1, four tensile specimens were machined from a cylinder which was cut

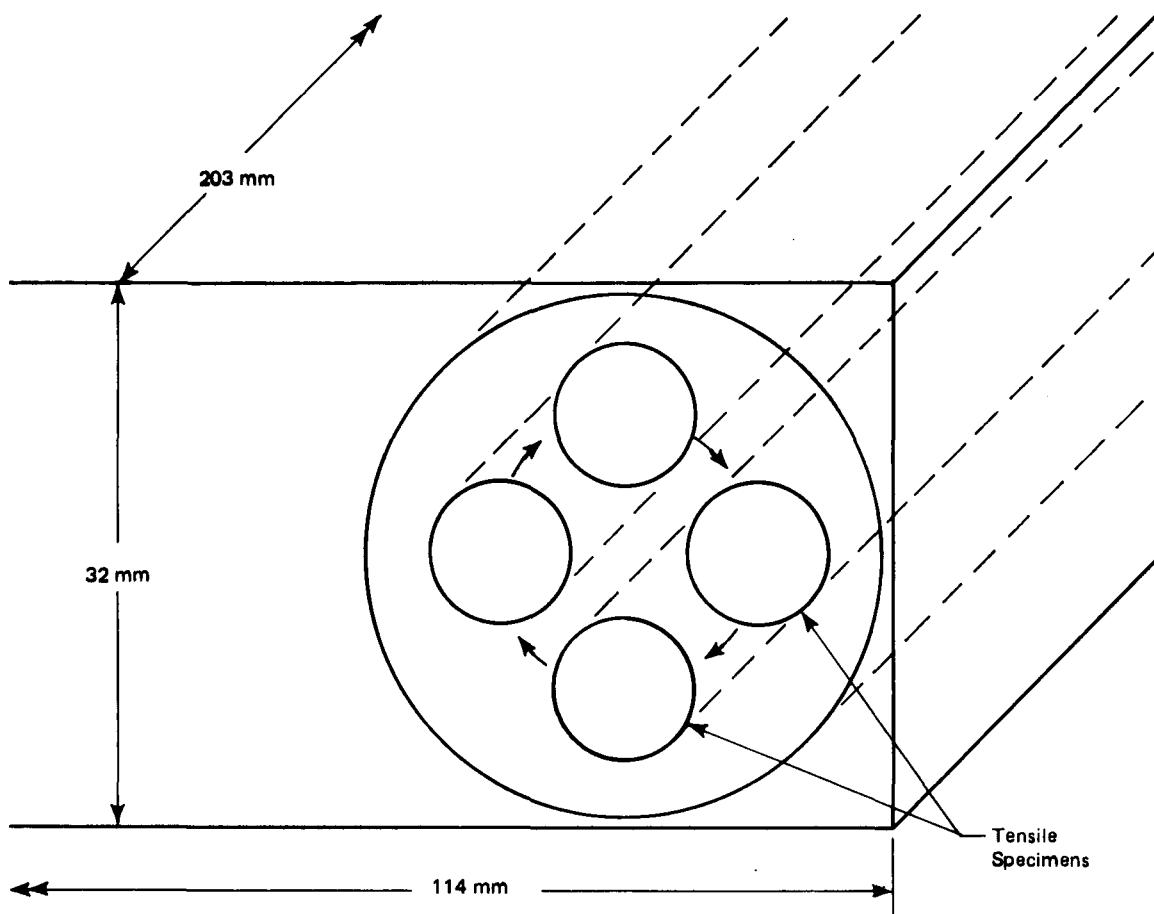


Figure 1. ORIENTATION OF TENSILE SPECIMENS RELATIVE TO THE PLATE FROM WHICH THEY WERE CUT.
(Exact Rotational Orientation of the Specimens was not Known)

(a) Performed under the direction of J. M. Jones of the Metal Preparation Division.

from one side of the plate. The tensile specimens were 51 mm long with a 4.1-mm-diameter, 16.3-mm-long gage length. The exact rotation of the specimens with respect to the plate is not known, as indicated in Figure 1, but was the causative factor that produced the variable elongations.

RESULTS

Table 1 lists the elongations, reductions in cross-sectional area, microhardness, and yield strengths measured on the four tensile specimens. The elongations varied between 9.4 and

Table 1
YIELD STRENGTHS, ELONGATIONS, MICROHARDNESSES, AND REDUCTIONS IN CROSS-SECTIONAL AREA MEASURED ON THE FOUR TENSILE SPECIMENS

Specimen Number	Yield Strength ⁽¹⁾ (MPa)	Elongation (%)	Reduction in Cross-Sectional Area			Microhardness (DPH) ⁽⁴⁾
			ΔA_1 ⁽²⁾ (%)	ΔA_2 ⁽³⁾ (%)	$\Delta A_1/\Delta A_2$	
1	0.999×10^3	17.2	17.4	7.4	2.4	494
2	1.046×10^3	15.6	13.7	10.1	1.4	496
3	1.029×10^3	9.4	10.5	3.6	2.9	478
4	1.054×10^3	9.4	7.5	2.4	3.1	492

(1) At 0.85% extension.

(2) Calculated at the fracture surface.

(3) Calculated at the end of the gage length.

(4) With a 500-gram load.

17.2%, and the reduction in cross-sectional area, ΔA , differed by a factor of at least 2.4 along the gage length of all specimens except Specimen 2, for which ΔA varied by only a factor of 1.4. To within experimental uncertainties, the microhardnesses of the specimens were the same. A correlation between the elongation and yield strength of the four specimens is not evident from the data listed in Table 1. For example, the elongations of Specimens 1 and 4 differed by a factor of 1.8, but their yield strengths only differed by 5.5%. Specimen 1, however, did have a significant amount of necking near the fracture surface, probably contributing to its higher elongation.

A scanning electron microscopy study^(b) showed that the fracture surfaces of the tensile specimens were similar; however, the two more-ductile specimens (Specimens 1 and 2) had more areas of dimple and stretch-type fracture formations, while the more brittle specimens

(b) Performed by R. K. Bennett, Jr of the Development Division.

(Specimens 3 and 4) had more cleavage and intergranular fracture areas. This result shows that the fracture mechanics differ in degree rather than in the kind of fracture.

Figure 2 presents photomicrographs taken from longitudinal sections of the specimens at the fracture surfaces^(c). Two types of microstructures are evident. Specimen 2 (View b) consists completely of lenticular plates; the other three specimens have duplex structures of lenticular-plate areas intermixed with almost featureless areas. Using two-stage replication techniques in transmission electron microscopy^(d) at magnifications of 52,500, the featureless areas were observed to have a pearlite-like structure. With thin-film techniques for electron microscopy, Eckelmeyer¹ found that the pearlite-like structure consisted of rows of 20 to 50-nm-diameter particles of U₂Ti in a matrix of the alpha phase. Eckelmeyer also showed that the lenticular-plate structure was the martensitic α' phase.

The duplex structure of Views a, c, and d (Specimens 1, 3, and 4) was not uniform along the longitudinal axis of these specimens. As the ends of the test specimens were approached, the amounts of lenticular-plate areas increased in proportion to the featureless areas. At their ends, Specimens 1 and 4 had completely lenticular-plate structures, similar to View b. Figure 3 emphasizes the change in the duplex structure of Specimen 3 from the end of the specimen toward the fracture surface. Even at its end, Specimen 3 did not have a completely lenticular-plate structure. On the other hand, the structure of Specimen 2 was completely lenticular plate along its length, as seen in Figure 2, View b.

Microhardness (\sim 500 DPH) and titanium concentrations^(e) (\sim 0.7%) of the featureless and lenticular-plate areas of Specimens 1, 3, and 4 were the same, to within experimental error, and were also the same as the lenticular-plate areas of Specimen 2. Neither iron nor silicon were found in the specimens by the electron microprobe.

(Limits of detection were 85 and 55 ppm, respectively.)

Table 2 lists the carbon and hydrogen contents of the specimens, which were determined from samples taken from the threaded regions of the tensile specimens. Both the carbon and hydrogen contents are well within acceptable limits and, therefore, could not account for the measured differences in ductility.

The ion microprobe^(f) was used to search for titanium concentration gradients in the specimens. To within the

Table 2

CARBON AND HYDROGEN CONCENTRATIONS IN THE SPECIMENS

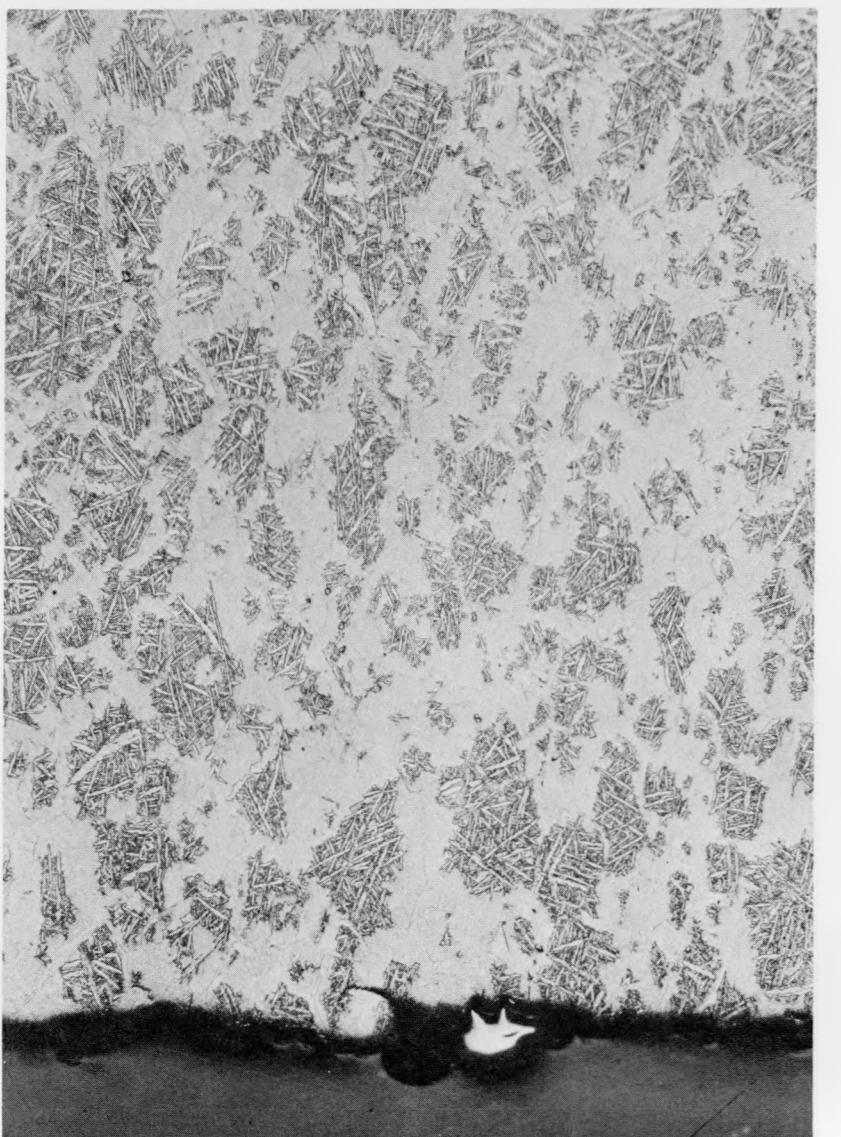
Specimen Number	Concentration (ppm)	
	Carbon	Hydrogen
1	16	0.08
2	19	0.09
3	20	0.09
4	23	0.08

(c) Photomicrographs and hardness measurements were made by H. C. East of the Product Certification Division and I. D. Conner of the Development Division.

(d) Electron microscopy performed by J. F. McLaughlin and S. H. Eary of the Development Division.

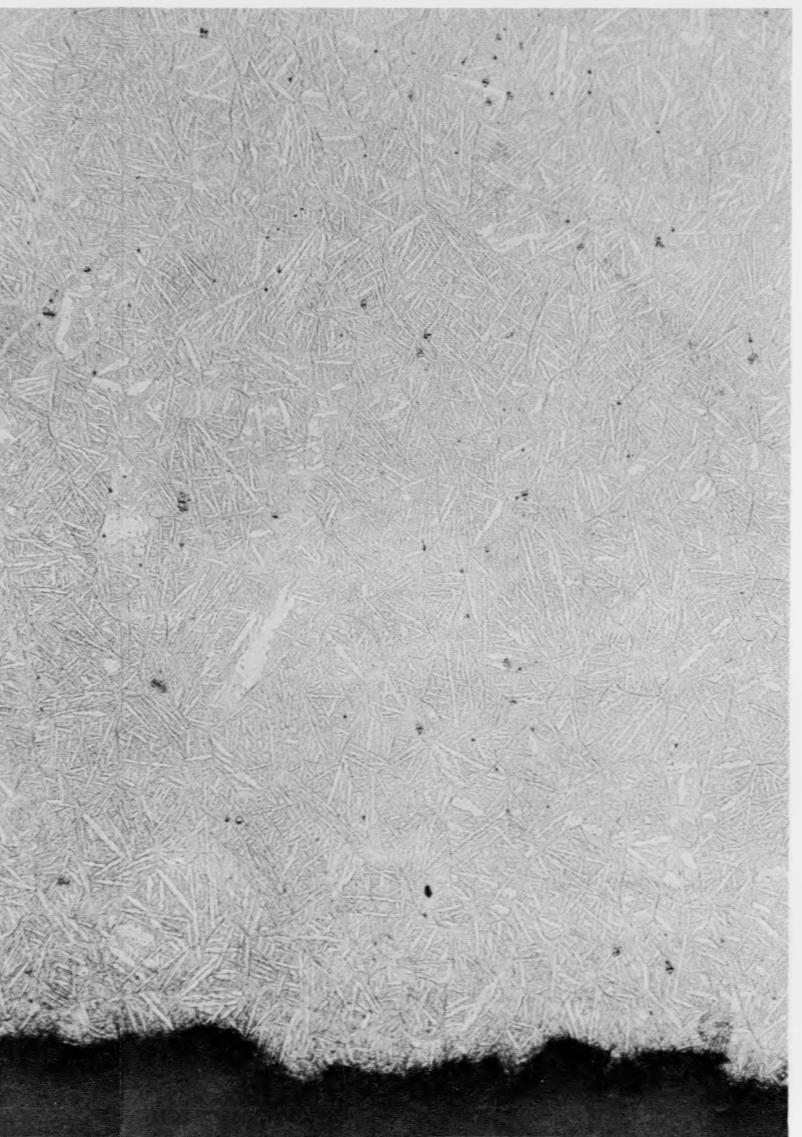
(e) Titanium concentrations were determined, using the electron microprobe, by L. R. Walker of the Product Certification Division. Weight percentage based solely on intensity ratios. Limit of sensitivity was approximately \pm 0.05%.

(f) Ion microprobe study performed by S. S. Cristy of the Development Division.



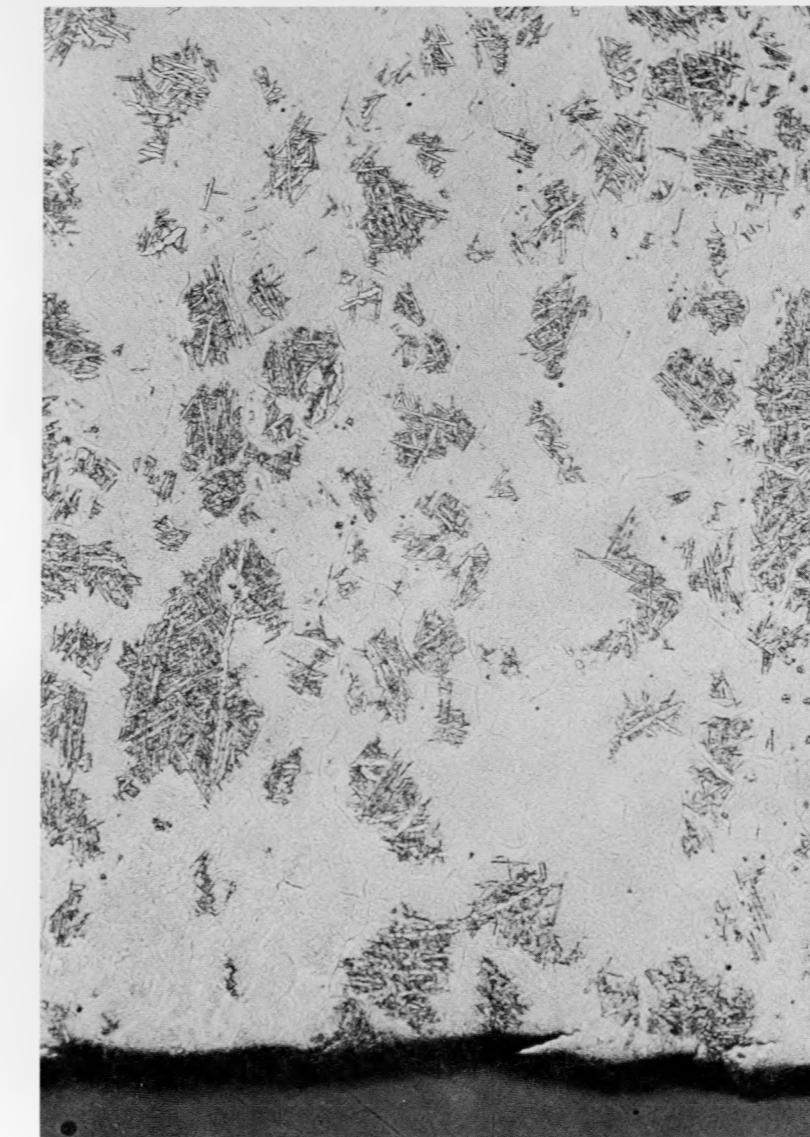
(a) Specimen 1. (duplex structure)

MS-77-0184-1



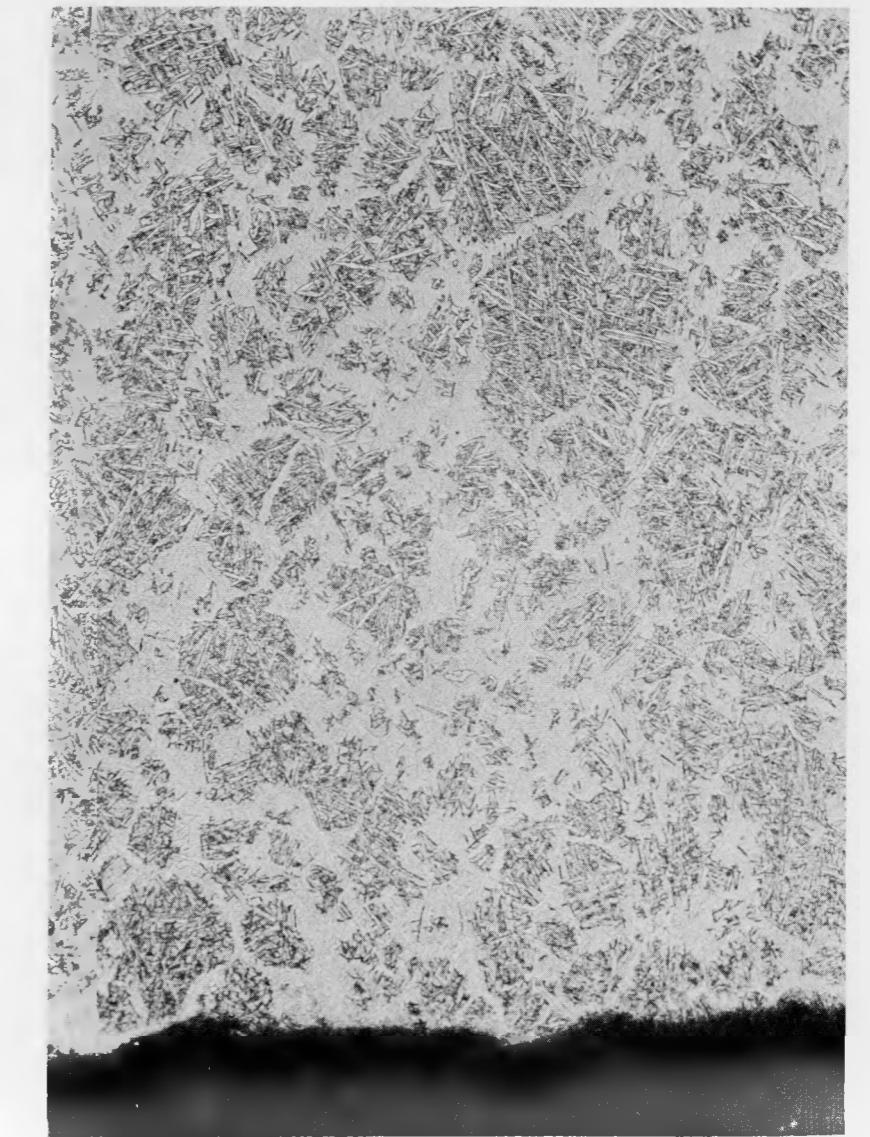
(b) Specimen 2. (lenticular-plate structure)

MS-77-0184-8



(c) Specimen 3. (duplex structure)

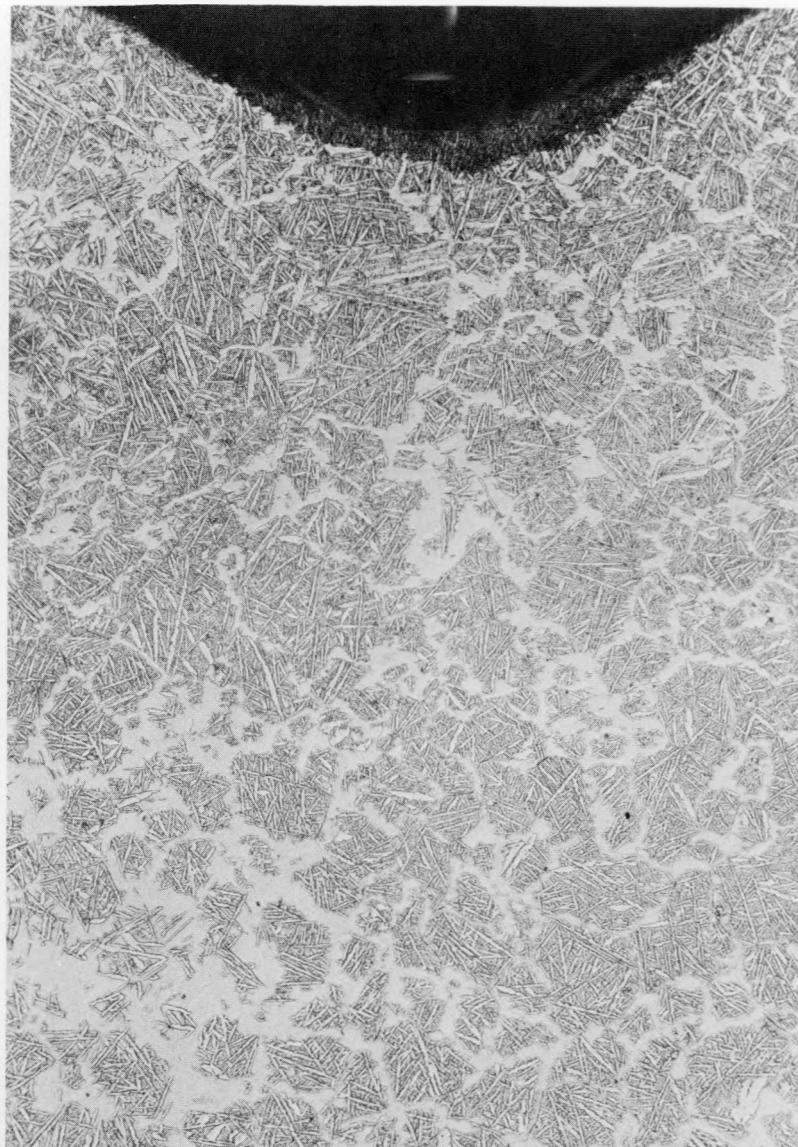
MS-77-0184-10



(d) Specimen 4. (duplex structure)

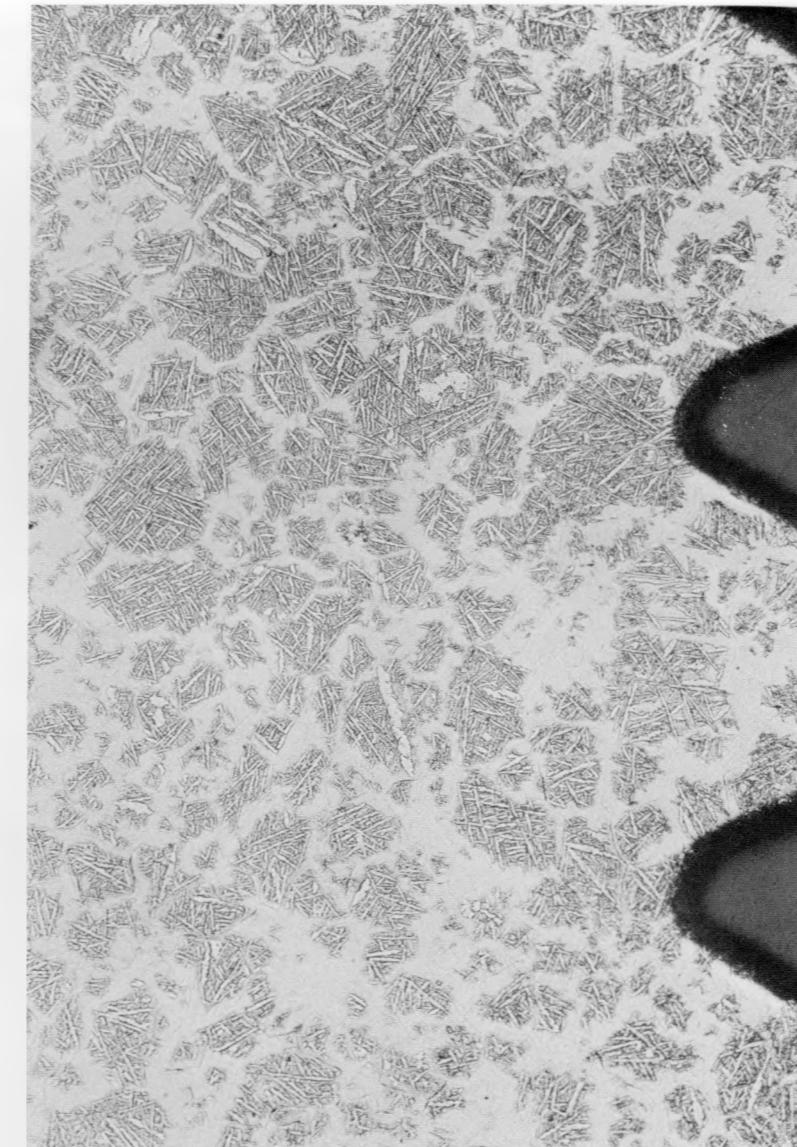
MS-77-0184-15

Figure 2. PHOTOMICROGRAPHS OF SPECIMENS AT THE FRACTURE SURFACE, SHOWING DUPLEX AND LENTICULAR-PLATE STRUCTURES. (50X)



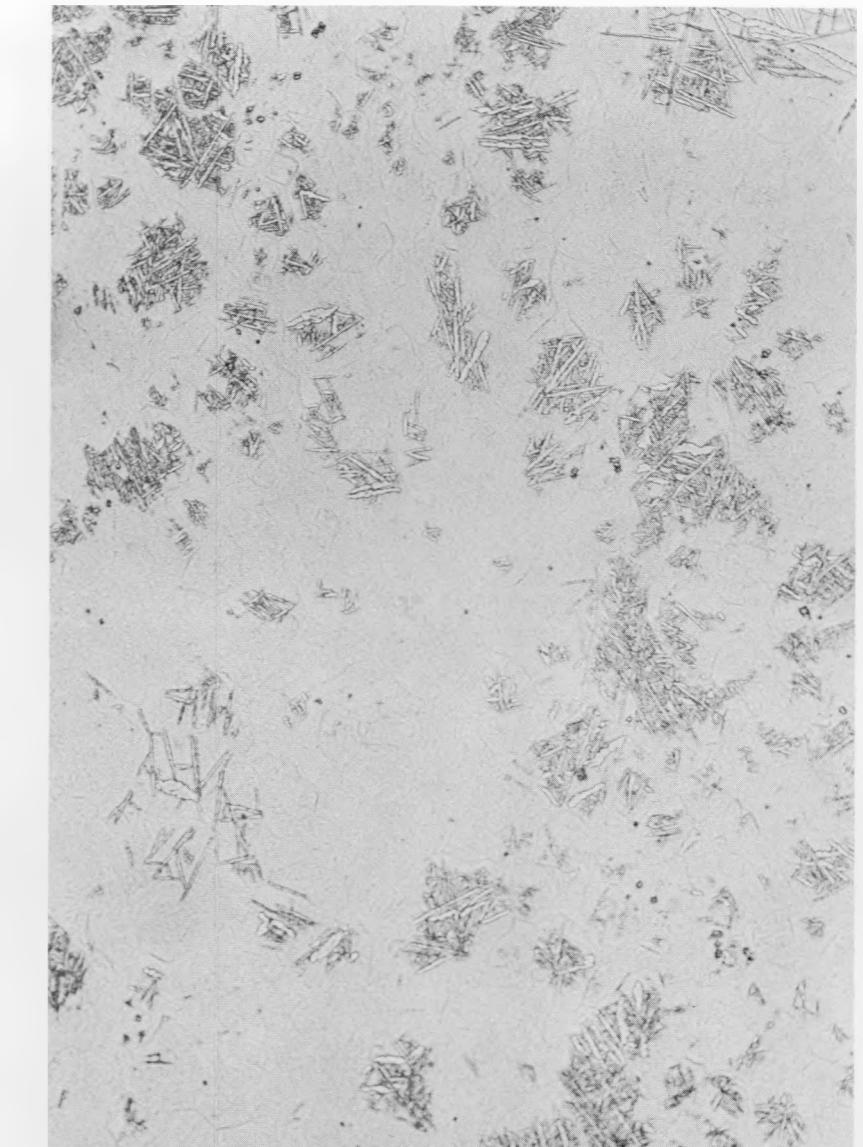
(a) End of the Specimen.

MS-77-0184-14



(b) Threaded Region of the Specimen.

MS-77-0184-13



(c) Gage-Length Region of the Specimen.

MS-77-0184-11

Figure 3. PHOTOMICROGRAPHS OF TENSILE SPECIMEN 3, SHOWING THE CHANGE IN THE DUPLEX STRUCTURE FROM THE END OF THE SPECIMEN TOWARD THE FRACTURE SURFACE. (50X)

beam size of 2 μm , the titanium was uniformly distributed in the four specimens. Titanium-rich inclusions were found uniformly distributed and in about the same concentration in all four specimens.

DISCUSSION

Other than the differences in microstructure shown in Figure 2, the various tests conducted in this study did not yield results that could explain the low and variable ductilities. The cause for these low and variable ductilities was deduced from a heuristic argument, based on the observed microstructures, the work of Eckelmeyer,^{1,2} observations of the water-flow patterns in the quenching bath, and heat-transfer calculations.

Eckelmeyer studied the effects of quenching rates from the gamma-stable temperature region on the mechanical properties and morphology of the U-0.75 Ti alloy. Figure 4 is a plot of the elongation of the aged alloy versus the quenching rate from Eckelmeyer's work. Between quenching rates of 50 and 50 $^{\circ}$ C/s, these data show that the elongation falls from 25 to 7%, respectively. Unfortunately, Eckelmeyer did not present comparable data for quenching rates greater than 50 $^{\circ}$ C/s.^(g) Eckelmeyer also showed that all the titanium is retained in solution in the α' martensitic phase if the quenching rate is greater than 200 $^{\circ}$ C/s in the critical temperature region (about 660 to 500 $^{\circ}$ C). Figure 5 is a plot of the percent of retained α' versus the quenching rate. By comparing Figures 4 and 5, it is apparent that the major reduction in elongation begins when the amount of retained α' falls below 50%, which occurs for quenching rates below 20 $^{\circ}$ C/s.

When viewed in an optical microscope, Eckelmeyer found that the α' martensitic phase had a lenticular-plate morphology. Also, the titanium precipitated as the compound U_2Ti in a matrix of alpha uranium, and the $\alpha + \text{U}_2\text{Ti}$ phase appeared "featureless" in the optical microscope. Only at approximately 100,000X in thin-film transmission electron microscopy could the pearlite-like structure of the $\alpha + \text{U}_2\text{Ti}$ mixture be resolved. According to Eckelmeyer, a duplex structure of lenticular plates and featureless areas in the optical microscope, such as in Figure 2, is a sure sign that the quenching rate from the gamma phase was below about 75 $^{\circ}$ C/s.

The quenching rates at the mutual center of the width and length of a 32-mm-thick plate were calculated by using an equation derived by Carslaw and Jaeger.⁴ To simplify the solution, one-dimensional heat flow in the direction of the thickness was assumed, the heat-transfer coefficient at the water/plate interface was assumed to be infinite, and the thermal diffusivity of U-0.75 Ti was assumed to be constant ($0.12 \times 10^{-4} \text{ m}^2/\text{s}$). Figure 6 presents plots of the quenching (cooling) rates versus temperature for various distances from

(g) It is conceivable that the elongation decreases as the quenching rate increases above 50 $^{\circ}$ C/s. If so, some of the variability in the elongation measurements on this alloy could be explained more easily. Future studies should investigate this point.

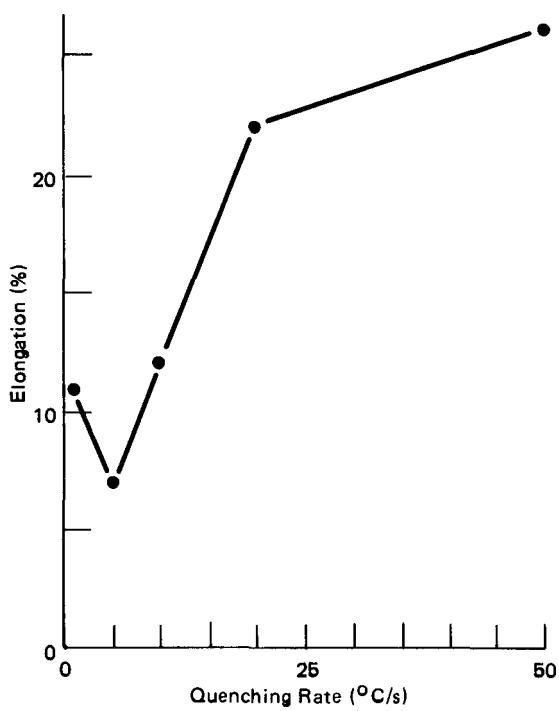


Figure 4. ELONGATION OF AGED URANIUM-0.75 TITANIUM ALLOY VERSUS THE QUENCHING RATE FROM THE GAMMA-PHASE REGION (800° C). (Obtained by Eckelmeyer for the U-Ti Alloy)

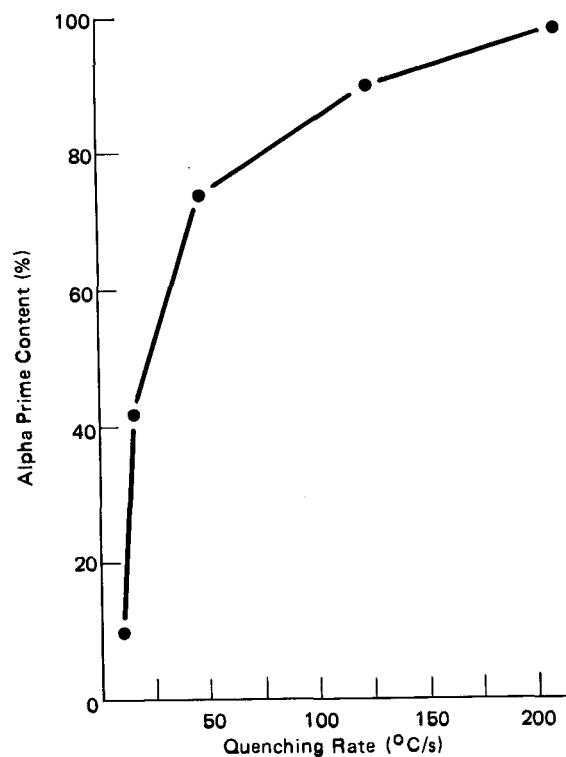
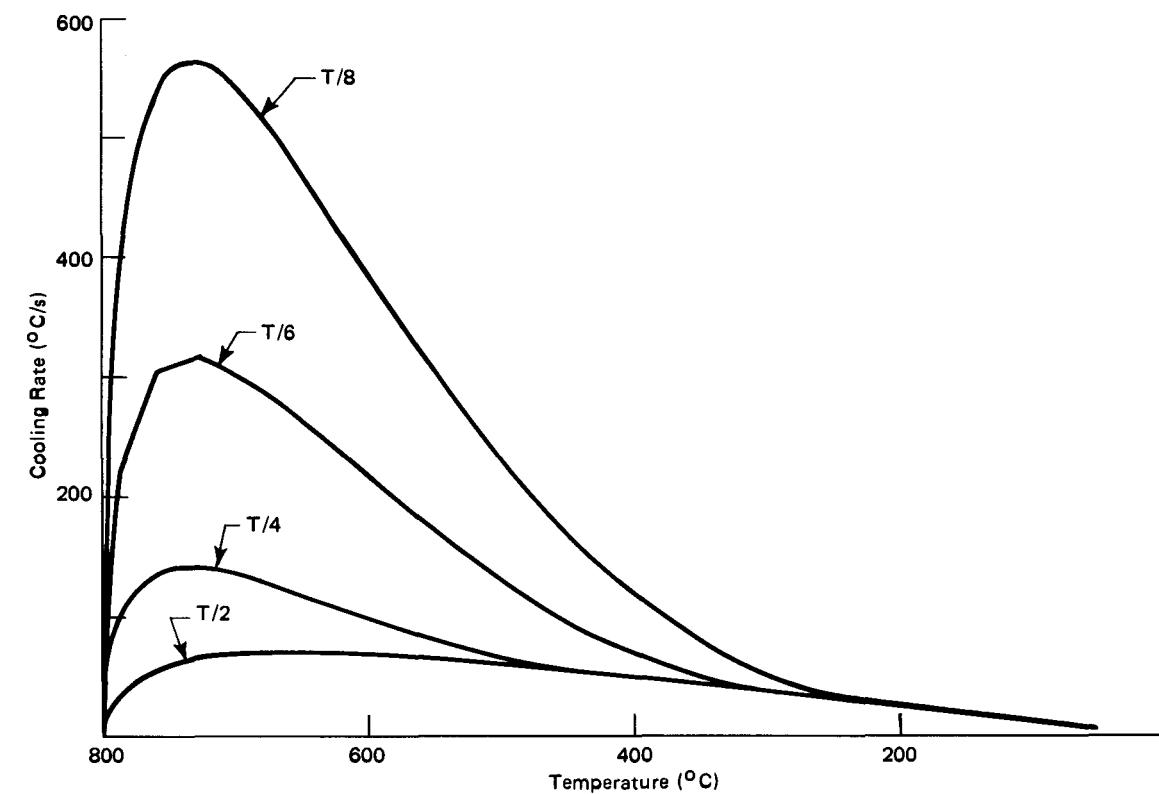


Figure 5. PERCENT RETAINED ALPHA-PRIME PHASE VERSUS THE QUENCHING RATE FROM THE GAMMA-PHASE REGION (800° C). (Obtained by Eckelmeyer for the U-Ti Alloy)

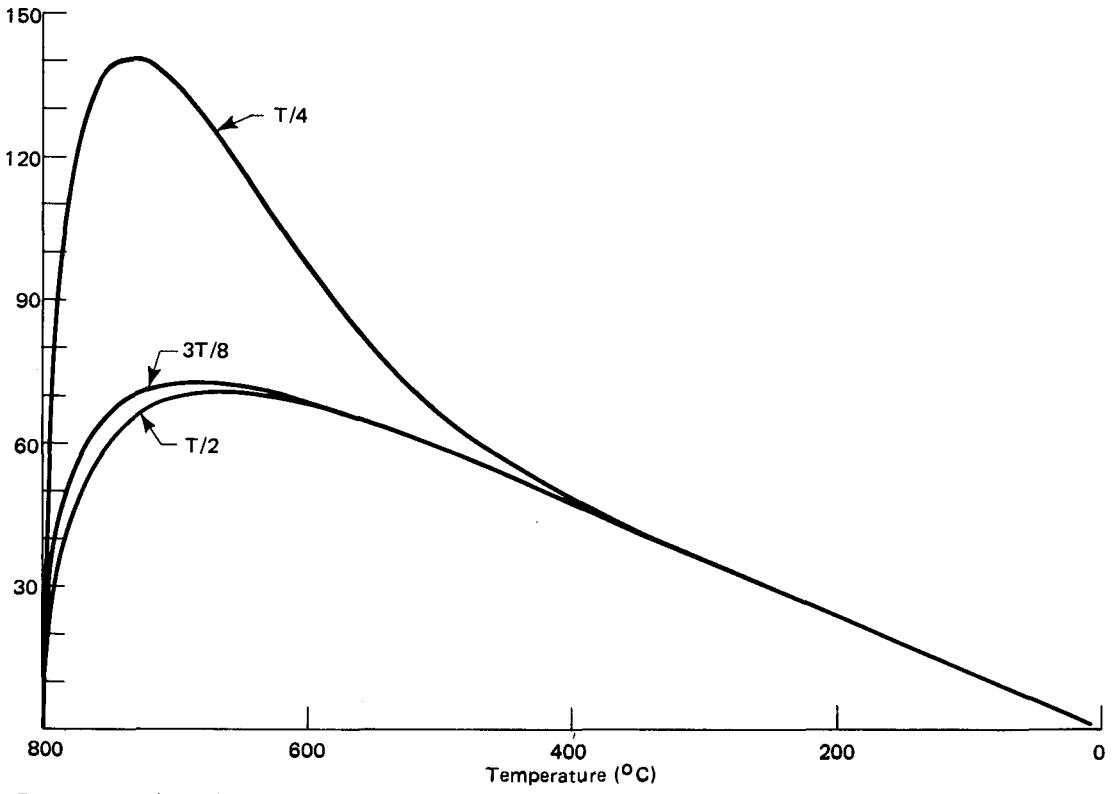
the face of the center of the plate. The quenching rate at the mid thickness ($T/2$) was about 60° C/s between 660 and 500° C, and the quenched rate was about 350° C/s at a depth of $T/8$, where T (32 mm) is the thickness of the plate.

The calculations of Figure 6 are "ideal" and probably represent the upper limit of the achievable quenching rates. Even then, complete retention of titanium would occur only at distances less than about 5 mm ($T/6$) from the face of a 32-mm-thick plate, and the mid thickness ($T/2 = 16$ mm) would have a structure of 75 to 80% α' . Undoubtedly, the quenching rates achieved in production are substantially less than the ideal values presented in Figure 6. Thus, the mid thickness of a 32-mm-thick production plate could, conceivably, have quenching rates well below 60° C/s and, therefore, tensile specimens taken from near the mid thickness would have low elongations ($\sim 10\%$). On the other hand, tensile specimens from near the face of a 32-mm-thick production plate would have higher elongations ($\sim 20\%$) because the quenching rates would be higher in that part of the plate. Recapitulating—variable elongations were caused by sampling at various distances from the face of the plate (Figure 1), whereas low elongations were due to poor heat-transfer conditions in the quenching bath.

Visual observations of the quenching bath showed that when the plate was out of the bath, adequate stirring existed in the bath, but when the plates were lowered into the bath, the rack holding the plates acted as a baffle, essentially becalming the water near the plates.



(a) Distances of $T/8$, $T/6$, $T/4$, and $T/2$, (where $T = 32$ mm) From the Face of the Plate.



(b) Distances of $T/4$, $3T/8$, and $T/2$ (where $T = 32$ mm) From the Face of the Plate.

Figure 6. CALCULATED QUENCHING (COOLING) RATES VERSUS THE TEMPERATURE AT THE MUTUAL CENTER OF THE WIDTH AND LENGTH OF A 32-mm-THICK PLATE OF URANIUM-0.75 TITANIUM ALLOY AS A FUNCTION OF THE DISTANCE FROM THE FACE OF THE PLATE.

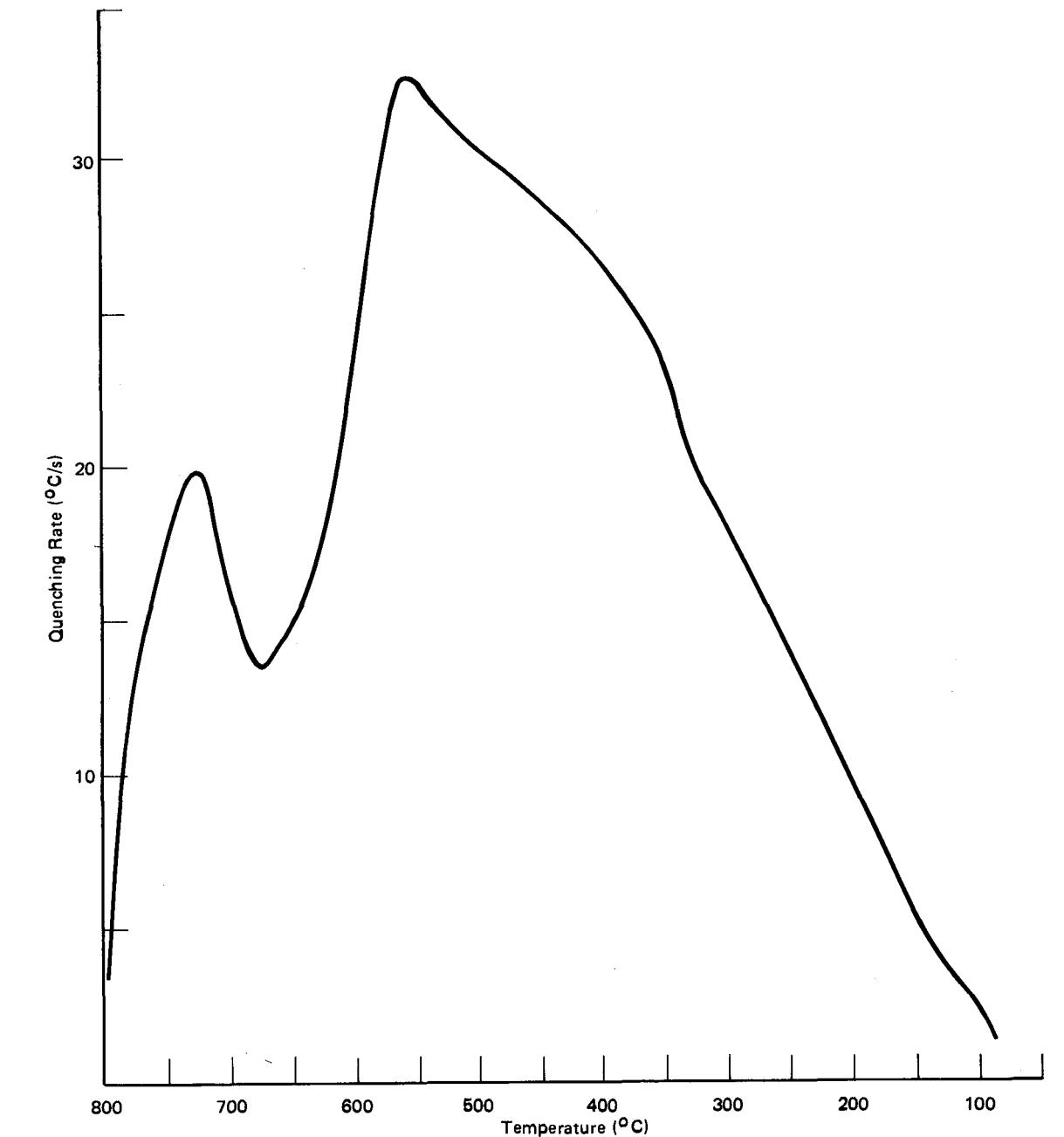


Figure 7. QUENCHING RATE VERSUS TEMPERATURE IN THE CENTER OF THE WALL THICKNESS OF A 210-mm-LONG, 147-mm-ID, 29-mm-WALL, HOLLOW CYLINDER OF URANIUM-0.75 TITANIUM ALLOY DURING QUENCHING. (Quenched from 800 $^{\circ}$ C in the Quenching Bath Used with the 32-mm-Thick Plate)

Without adequate water movement, a steam/vapor layer forms around the plate during quenching, drastically reducing heat transfer between the plate and the water. It is postulated that poor heat transfer between the plate and the water of the quenching bath resulted in low quenching rates, yielding low elongations in specimens taken from the center of the plate. Unfortunately, the exact orientation of the four tensile specimens with respect to the centerline of the plate was not known (Figure 1).

Figure 7 is a plot^(h) of the quenching rate versus temperature in the center of the wall thickness of a 210-mm-long, 147-mm-ID, 29-mm-wall, hollow cylinder of U-0.75 Ti during quenching from 800° C in the same quenching bath used in this study. While it is realized that heat transfer from a hollow cylinder differs from that of a plate, these data are a first approximation to the quenching rates in the plate. Note that the quenching rates reached a minimum near 660° C because the $\gamma \rightarrow \alpha + U_2Ti$ transformation is exothermic. Also, the maximum quenching rate of 30° C/s occurred near 550° C.

The data of Figure 7 and the visual observations of mixing in the quenching bath support the hypothesis that the low elongations in the plate were caused by low quenching rates due to poor heat-transfer conditions in the quenching bath.

CONCLUSIONS AND RECOMMENDATIONS

It was concluded that the variable and low elongations in four specimens from the same gamma-quenched-and-aged plate of U-0.75 Ti was due to poor heat-transfer conditions in the quenching bath and the method of sampling for the tensile specimens. Two solutions to the problem are suggested: (1) add a third element to the alloy to slow down the $\gamma \rightarrow \alpha + U_2Ti$ transformation so that titanium is retained in solution in the martensitic α' phase, or (2) achieve a substantial improvement of the heat-transfer coefficients in the quenching bath. The latter proposal is the near-term solution whereas the former proposal is the long-range and better solution. It is also recommended that the orientation of the tensile specimens with respect to the centerline of the plate be documented.

(h) These data were obtained by P. S. Lewis, Jr, H. L. Wigginton, and L. C. Howington of the Development Division.

REFERENCES

1. Eckelmeyer, K. H.; *The Effects of Heat Treatments on the Microstructure and Mechanical Properties of U-0.75 Wt % Ti*, SAND 75-0599; June 1976.
2. Eckelmeyer, K. H. and Zanner, F. J.; *J Nuc Matls* 67, p 33 (1977).
3. Ammons, A. M.; "Precipitation Hardening in Uranium-Rich Uranium-Titanium Alloys", *Physical Metallurgy of Uranium Alloys*, p 511; Edited by J. J. Burke, et al; Brook Hill (1976).
4. Carslaw, H. S. and Jaeger, J. C.; *Conduction of Heat in Solids*, p 97; Clarendon Press, Oxford (1959).

Distribution**Lawrence Livermore Laboratory**

Wood, D. L.

Oak Ridge Y-12 Plant

Anderson, R. C.
Banker, J. G.
Beck, D. E.
Fraser, R. J.
Jessen, N. C., Jr
Johnson, D. H.
Jones, J. M.
Keith, A.
Kite, H. T.
Koger, J. W.
Kollie, T. G. (3)
Lewis, P. S., Jr
Martin, W. R./Dodson, W. H./Googin, J. M.
Mills, J. M., Jr/DOE-TIC (28)
Northcutt, W. G., Jr
Wigginton, H. L.
Y-12 Central Files (master copy)
Y-12 Central Files (route copy)
Y-12 Central Files (Y-12RC)
Y-12 Central Files (4)

Sandia - Albuquerque

Eckelmeyer, K. H.

Sandia - Livermore

Mote, M. W., Jr