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by

Charles A. Javorsky

U-0.83 wt% Ti ALLOY: COOLING RATES OF GAMMA-QUENCHED ALLOY, RESULTING MICROSTRUCTURE AND RESPONSE TO AGING

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ABSTRACT

Samples from a U-0.83 wt% Ti alloy were subjected to various cooling rates, and response to subsequent aging was investigated. By a suitable choice of quenching media, cooling rates were varied from 3.9 to 190°C/sec. Resulting as-quenched microstructures and hardnesses were not a function of cooling rates when rates were greater than 40°C/sec. Aging treatments were monitored by metallographic observations and hardness measurements, and cooling rates greater than 40°C/sec produced no relative changes in aging response. Tensile properties of selected quenched and aged specimens indicate that strength and ductility are unaffected by cooling rate.

I. INTRODUCTION

Dilute uranium-titanium alloys possess a useful range of mechanical properties which can be developed by quenching from the high-temperature gamma phase, with subsequent artificial aging in the alpha-plus-delta phase region. At room temperature, the as-quenched martensitic structure consists of a distorted crystal lattice, commonly referred to as alpha prime. The aging process results in a submicroscopic coherent precipitate of delta-phase U_2Ti within the alpha-prime lattice, and this precipitate produces increased hardness and strength in the alloy.

Some inconsistent aging behavior of these alloys results in variations in hardness, strength, and ductility. One possible explanation could be variations in cooling rates caused by mass effects or quenching techniques from the gamma phase. The purpose of this investigation was to relate, if possible, the cooling rates of dilute uranium-titanium alloys quenched in various media to the subsequent aging response in each case.

II. EXPERIMENTAL PROCEDURE

A casting of 1-in. diam and 26-in. length was prepared. It had a composition of U-0.83 wt% Ti, center and bottom, as determined by chemical analysis. Complete spectrochemical analyses indicated that the trace impurities were typical of good quality uranium alloy (see Table I).

TABLE I

CHEMICAL AND SPECTROCHEMICAL ANALYSIS OF U-0.83 wt% Ti CASTING

Constituent	Hot Top (ppm)	Center (ppm)	Bottom (ppm)
C	780	80	80
Ti	(1.22%)	(0.83%)	(0.83%)
Al		7	
Si		100	
P		<100	
Fe		30	
Ni		20	
Cu		15	
Sr		<40	
Mo		<25	

The complete casting, including the hot top, was machined to 0.875-in. diam and radiographed. The bottom 3 in. of the cast rod was discarded because of some centerline porosity. The hot top contained an excessive amount of titanium (Fig. 1), mostly in the form of TiC, and was also discarded. The remainder of the casting had a typical as-cast alpha-plus-delta microstructure which was free of defects (Figs. 2 and 3). A hardness survey of the center and bottom of this rod showed uniform hardness values of 320 DPH.

The specimens used for the cooling-rate experiments were machined from the central portion of the as-cast rod and were 0.875 in. in diameter and 0.845 in. long. An axial hole of 0.120-in. diam was bored to the geometric center of each specimen, and an intersecting thermocouple anchor hole was drilled near one end, as shown in Fig. 4. The hollow ceramic anchor tube was used also for suspending the specimen within the vertical tube furnace.

Cooling curves were obtained using a high-speed Brown time-temperature recorder and Pt-Pt 10-Rh thermocouples. The vertical Lindberg furnace contained a nickel tube with end caps which helped to maintain a uniform temperature zone during the heating in flowing argon. The specimen temperature was monitored on the recorder during solution heating at 830°C for 2.5 h; however, the time axis of the

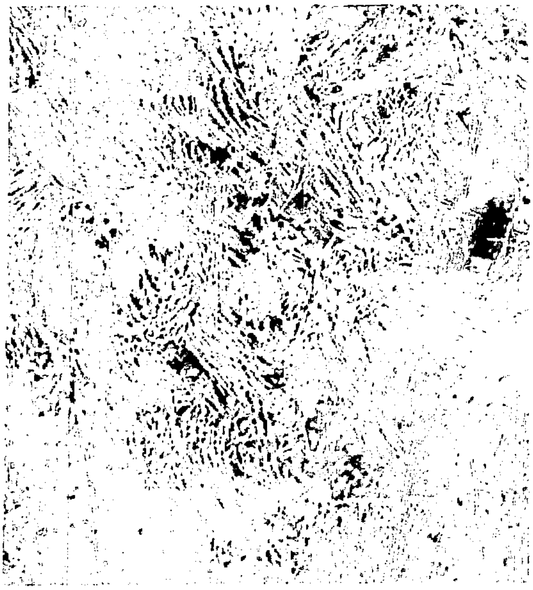


Fig. 2. Typical as-cast microstructure of U-0.83 wt% Ti alloy cast rod in centerline region (100X).

Fig. 1. Typical as-cast microstructure of U-0.83 wt% Ti alloy cast rod in hot-top region (100X).

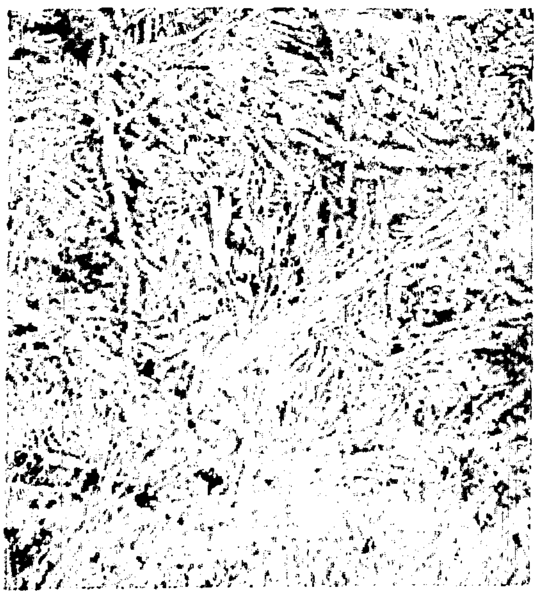


Fig. 3. Typical as-cast microstructure of U-0.83 wt% Ti alloy cast rod in bottom region (250X).

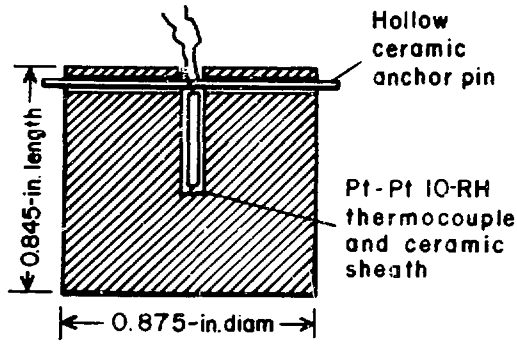


Fig. 4. U-0.83 wt% Ti cooling-rate specimen.

recorder was not activated until the specimen was released. The quenched specimens were then sectioned near the thermocouple tip, and microstructures and hardness values were determined. All samples were prepared with SiC papers and were given a final polish with 1-μ diamond paste on Met Cloth. A 10% oxalic acid electrolyte was used for etching at an open circuit voltage of 5 V dc.

Four specimens, one each quenched in brine, tap water, oil, and soluble oil, were chosen for aging-response determination. A sheathed Chromel-Alumel thermocouple was placed in contact with the four samples during aging at 342°C in flowing argon. At 6-h intervals during the aging treatment the samples were removed, air-cooled, and their hardnesses and microstructures determined. After a total aging time of 18 h at 342°C, the samples were given a final aging treatment of 3 h at 380°C.

Four tensile-test rods of 0.625-in. diam and 3-in. length were then machined from the remaining cast rod. These blanks were heated for 2 h and quenched from 840°C into oil or tap water to provide specimens at two quenching rates. The rods were subsequently aged for 3.5 h at 381°C in the same Lindberg tube furnace under flowing argon. Standard tensile-test specimens were then machined from the heat-treated blanks and, after testing, metallography specimens were cut from the butt ends of the broken samples. Six additional tensile-test rods consisting of as-cast U-0.54 wt% Ti were also heated for 2 h at 840°C and quenched into oil or tap water. These tensile specimens were then tested in the as-quenched condition.

All tensile-test specimens were tested using double strain gauges at a crosshead speed of 0.005 in./min through yield, then at 0.05 in./min to failure. Test temperature was 26°C, with 37% RH.

III. DISCUSSION AND RESULTS

Cooling rates were obtained by quenching the specimens in 10% brine, tap water, 0.05% polyvinyl alcohol (PVA), Super Gulf 70 oil, or 90% water-soluble oil, and aging them in flowing argon. The PVA presumably would yield a cooling rate between those of tap water and oil; and the soluble oil would give a rate slower than that of conventional oil.* The cooling-rate curves are plotted in Fig. 5 and show the various rates obtained between 830 and 600°C. (Values from 600-160° are extrapolated.) Since the eutectoid reaction, or alpha prime, for rapid cooling occurs at about 720°C, the cooling rate near that temperature should control the properties of the quenched alloy at room temperature.

The cooling rates shown in Table II and plotted in Fig. 5 were obtained from standard specimens which were 0.875 in. in diameter and 0.845 in. long. The specimen that was cooled in argon was water-quenched from 650°C to prevent aging in case any martensitic structure had been retained.

The DPH of the as-quenched specimens is about the same for each medium, except that the specimen quenched in argon has a lower hardness of 335 DPH. An average of 345 DPH is typical for a U-0.83 wt% Ti alloy (see Table III).

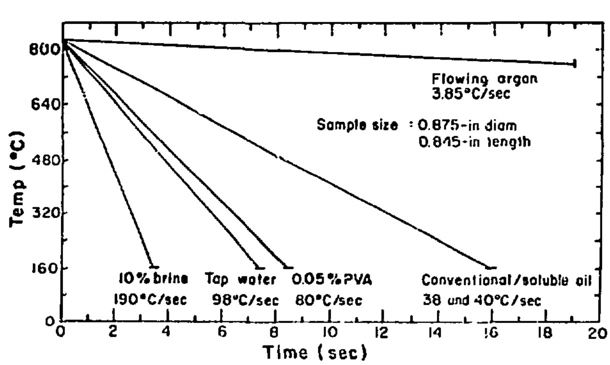


Fig. 5. Cooling rates of center region of U-0.83 wt% Ti in various media (no agitation).

*"Quenching of Steel," in ASM Metals Handbook (ASM, Metals Park, Ohio, 1964), 8th Ed., Vol. 2, pp. 15-36.

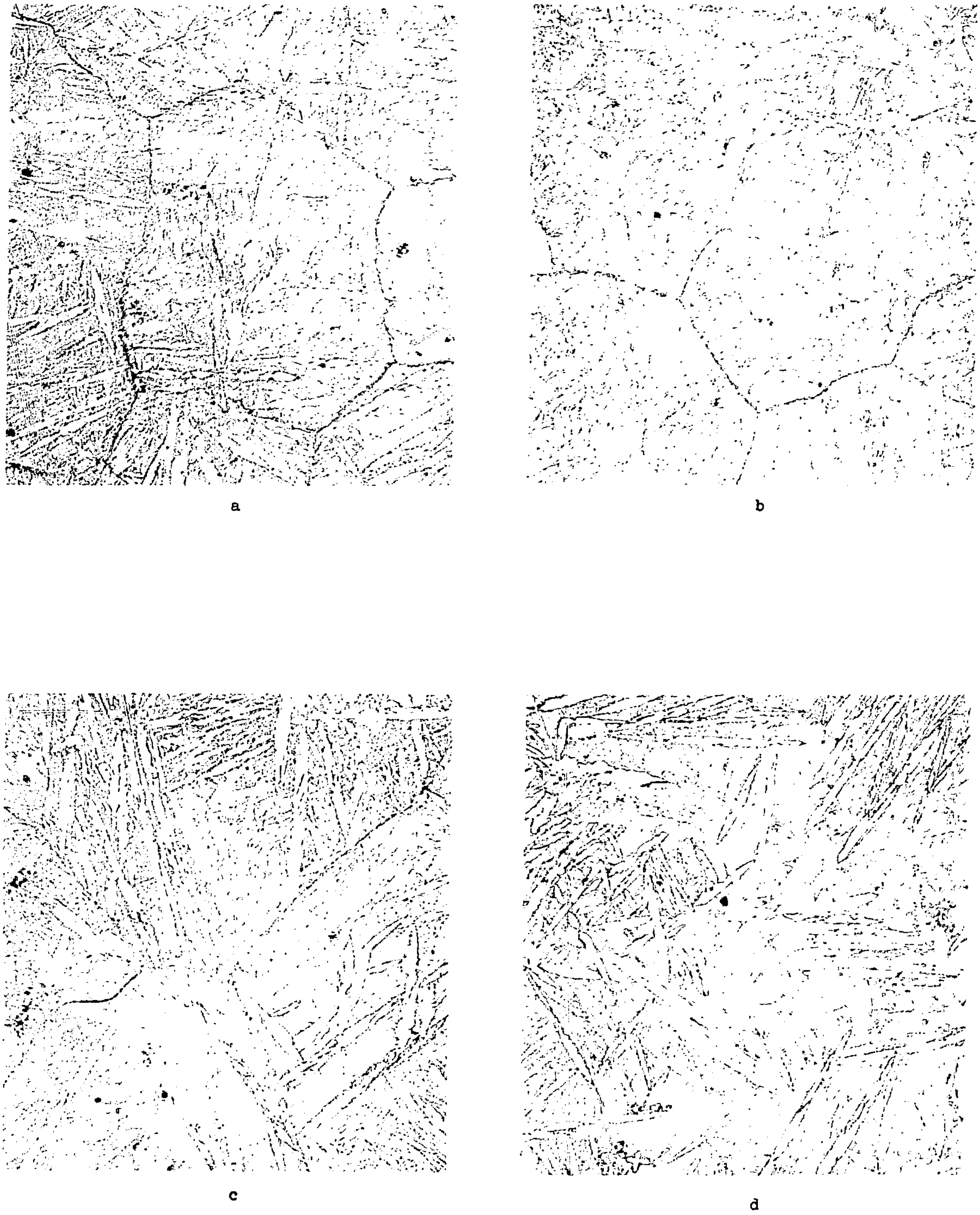
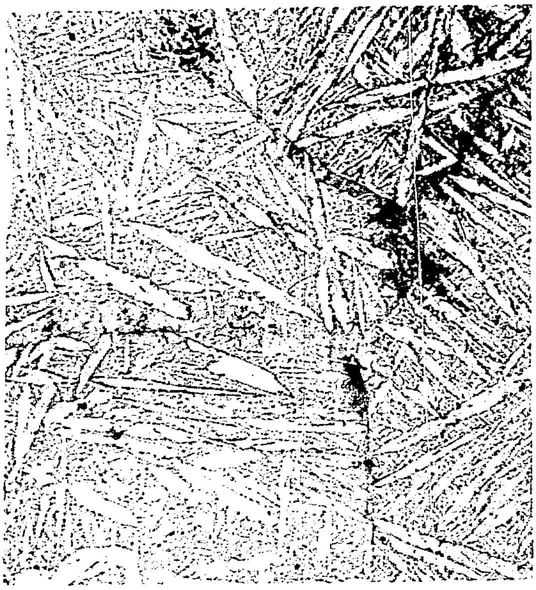
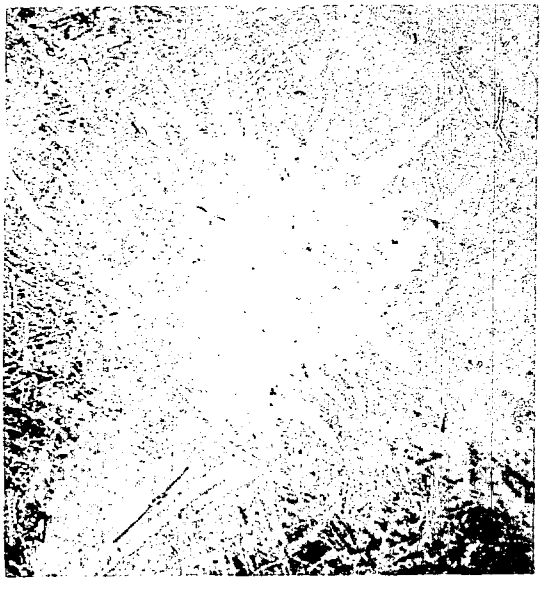


Fig. 6. Microstructure (250X) of as-quenched U-0.83 wt% Ti alloy taken at sample center. Quenching media: (a) 10% brine; (b) tap water; (c) 0.05% PVA, (d) oil.



e

f



g

Fig. 6 (cont). (e) 90% soluble oil; (f) and (g) flowing argon.

TABLE II

COOLING RATES OF QUENCHED U-0.83 wt% Ti ALLOY

Quench Medium	Center Cooling Rate (°C/sec)
10% NaCl (in water)	190
Tap water	98
0.05% PVA (in water)	80
Super Gulf 70 oil	38
90% soluble oil (in water)	40
Flowing argon in furnace	3.9

The microstructures of the as-quenched specimens are slightly dissimilar, particularly between samples quenched in oil at a rate of 38 to 40°C/sec and those quenched in water or brine at 98 and 190°C, respectively. Figure 6 illustrates the typical acicular matrix structures found at the sample centers. The grain boundary phase evident in the matrix of the oil-quenched specimens (Fig. 6d) is apparently formed by nucleation and growth but does not affect the hardness of the specimens. The grain boundary phase of the oil-quenched samples is similar to the fine structure evident within the acicular matrix of the more rapidly cooled samples.

The argon-cooled specimen (Figs. 6f and g) has a matrix of approximately 95% transformed alpha-plus-delta, typical of as-cast dilute uranium-titanium alloys. However, the slow cooling rate still produces acicular islands of martensitic structure surrounded by a transitional phase zone (Fig. 6g). Hardness across these transitional zones is uniform at 335 DPH--the same as for the remaining alpha-plus-delta matrix. It is evident that even at a slow cooling rate of 3.9°C/sec some high-temperature alpha-prime structure is retained, indicating that the uranium-titanium gamma-to-alpha phase change is rather sluggish.

To discount the possibility that the argon-cooled specimen possessed alloy segregation, the same specimen was reheated for 3 h at 840°C and argon-cooled to 650°C at the same rate of 3.9°C/sec, and was water-quenched as before. The microstructure and hardness values remained the same.

Of the four quenched specimens used for aging response, one each was quenched in brine, tap water, oil, or soluble oil. In all four cases the hardness increased by the same amount after aging for 6 h at 342°C (Table III), and the microstructures (Fig. 7) were unchanged from those of the as-quenched condition (Figs. 6a, b, d, e). The same aged samples were recycled for two more 6-h intervals at 342°C to see if any relative changes would occur. Hardness increased slightly--to 380 DPH--for all four samples (Table III). The microstructures examined after 18 h of aging showed no relative change from the as-quenched condition.

We concluded that the U-0.83 wt% Ti alloy would not show any relative age-hardening differences at 342°C after extended heating; therefore, the samples were given an additional 3-h treatment at 380°C so that we could examine aging behavior at a higher temperature. As shown in Table III, the hardness of the four samples increased to 400-405 DPH. The microstructures were examined after the final aging treatment and are illustrated in Fig. 8. The grain boundaries of the brine-quenched sample evidenced a slight change in structure; otherwise its matrix was similar to that of the as-quenched condition.

A plot of the aging response for the various heat treatments is shown in Fig. 9. The cooling

TABLE III

HARDNESS VALUES FOR AS-QUENCHED AND AGED U-0.83 wt% Ti ALLOY AS A FUNCTION OF QUENCH RATE

	10% Brine 190°C/Sec (DPH)	Tap Water 98°C/sec (DPH)	0.05% PVA 80°C/sec (DPH)	Oil 38°C/sec (DPH)	Soluble Oil 40°C/sec (DPH)	Flowing Argon 3.85°C/sec (DPH)
As-quenched	345	350	340	345	345	335
Aged 6 h at 342°C	370	367	--	375	375	--
Aged 6+6 h at 342°C	375	375	--	370	385	--
Aged 6+6+6 h at 342°C	380	380	--	375	375	--
Final 3 h at 380°C	405	400	--	400	405	--

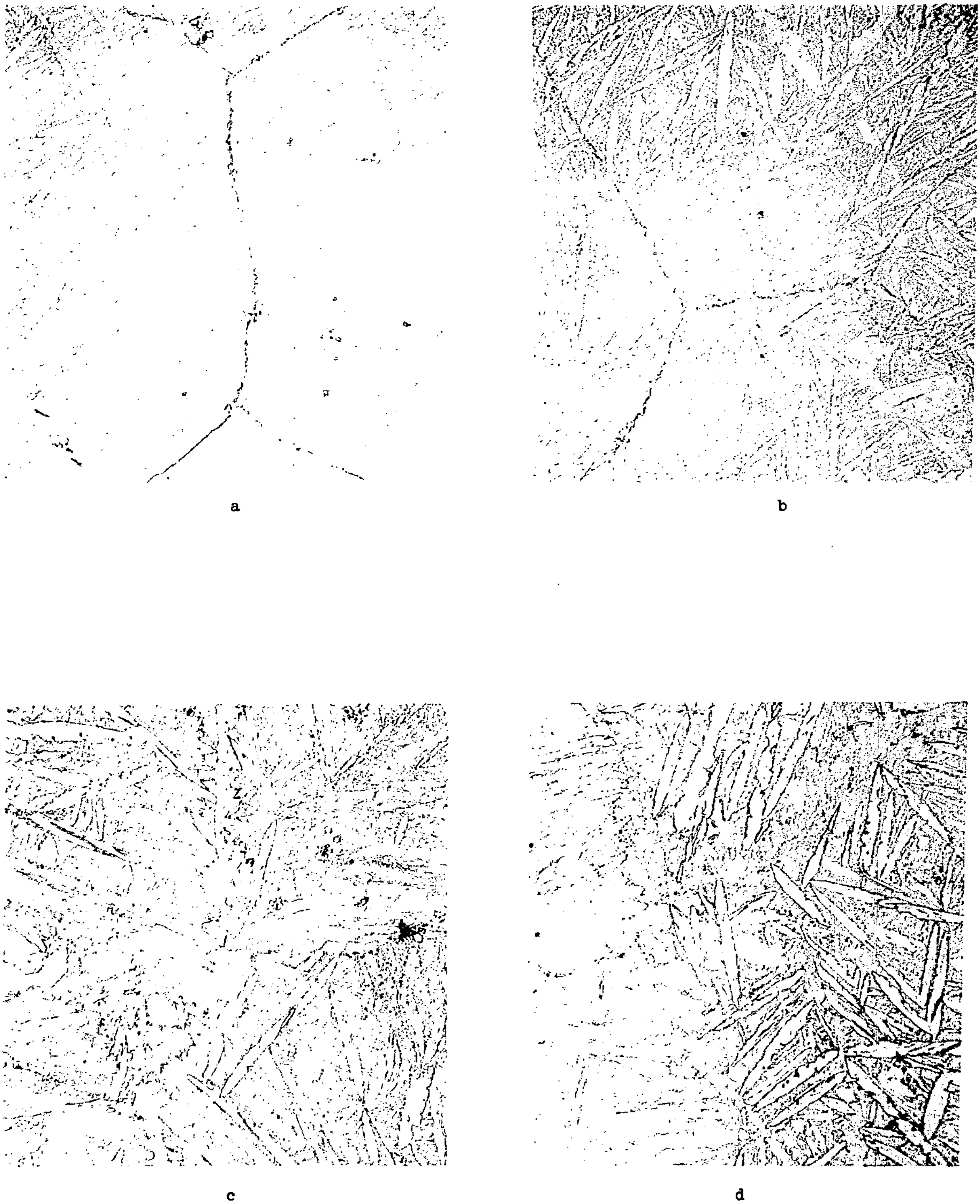
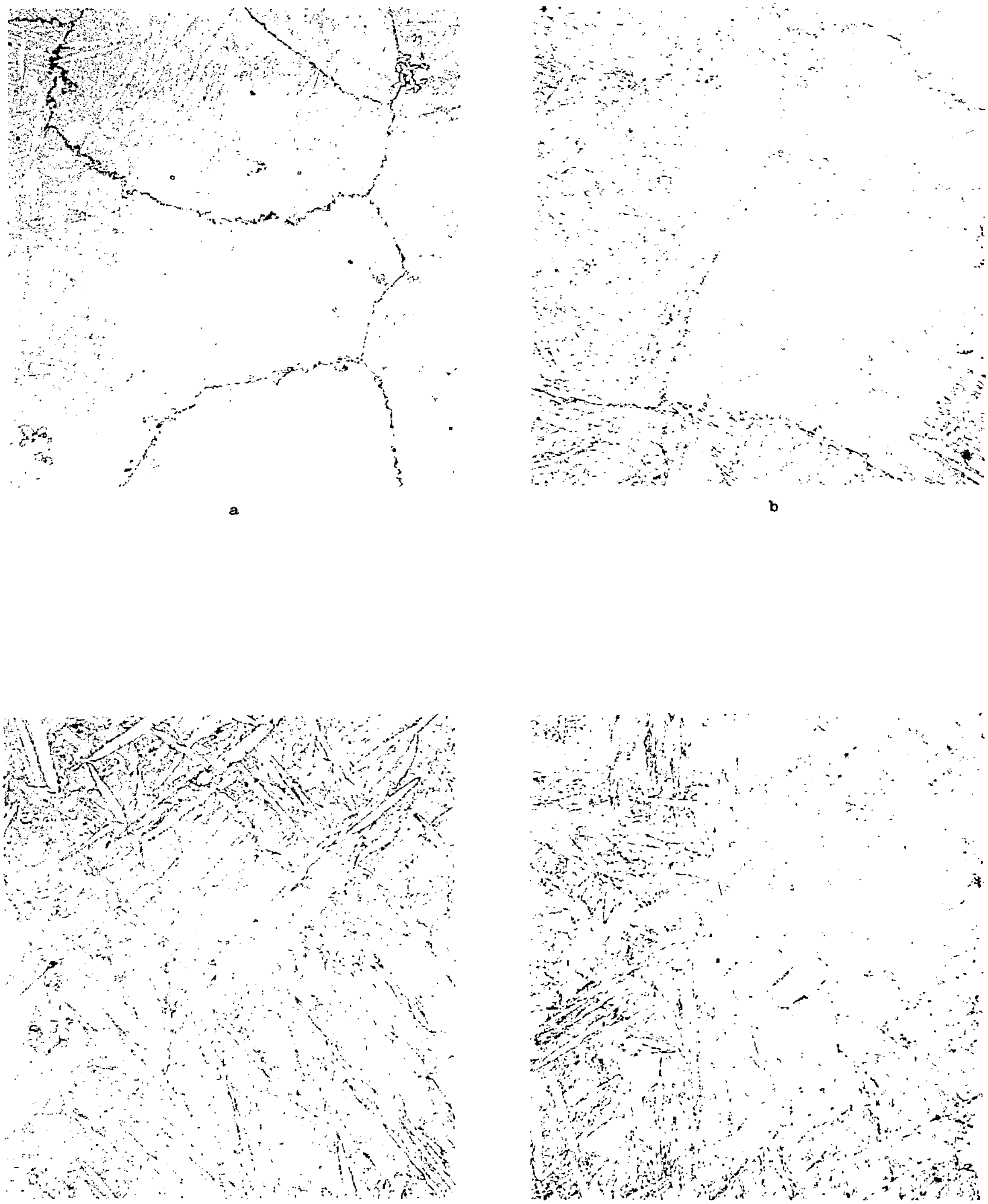


Fig. 7. Microstructure (250X) of samples aged for 6 h at 342°C. Quenching media: (a) 10% brine; (b) tap water; (c) oil; (d) soluble oil.



a b c d

Fig. 8. Microstructure (250X) of samples aged for 18 h at 342°C and then for 3 h at 380°C. Quenching media: (a) 10% brine; (b) tap water; (c) oil; (d) soluble oil.

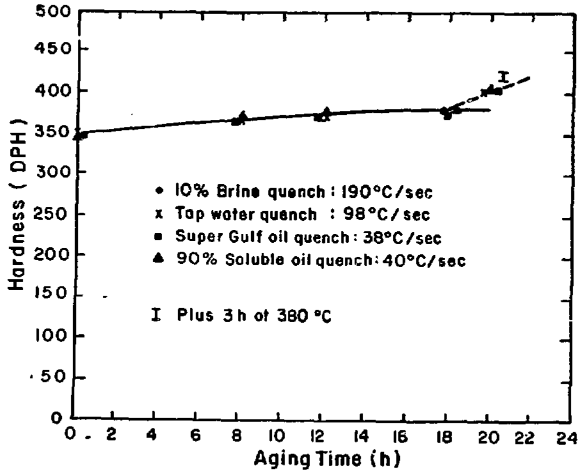


Fig. 9. Aging response of quenched samples after various heat treatments.

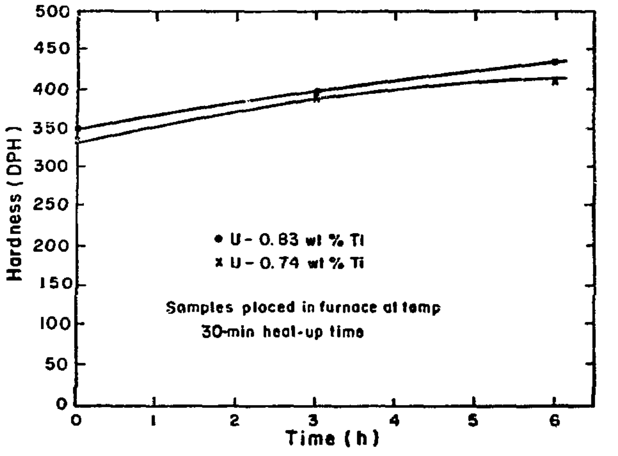


Fig. 10. Aging response of two uranium-titanium alloys heated at 380°C for two 3-h intervals.

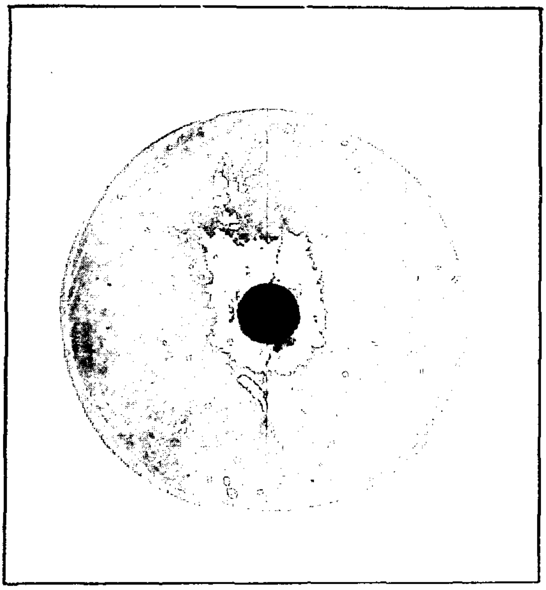
rates of brine and oil produced virtually no change in hardness in aged specimens. The data show that a cooling rate of 40°C/sec, or more, will produce consistent quenched and aged properties in these uranium-titanium alloys. Evidently water-quenching of these alloys will produce satisfactory aging response for a sample section of 0.875-in. diameter. Inconsistent mechanical properties normally should not be attributed to water quenching.

To see if the duplex aging treatment of 18 h at 342°C plus 3 h at 380°C would produce a hardness different from that produced by a single treatment at 380°C for only 3 h, a sample from the U-0.83 wt% Ti casting and a U-0.74 wt% Ti sample were quenched together into tap water and aged at 381°C for 3 h. The resulting hardnesses were 395 and 390 DPH, respectively (Fig. 10). Thus the initial low-temperature aging treatment of 18 h does not have a significant effect upon samples which are subsequently aged at 380°C. In fact, aging response is very temperature-dependent. The two samples were given an additional aging treatment of 3 h and the aging curves of Fig. 10 show that aging response varies with the concentration of titanium in the sample.

The brine-quenched alloy had severe cratering and cracking in the region of the thermocouple hole. The nature of these centerline defects can be seen in a transverse section taken from the base of the hole (Fig. 11). A brine quench is obviously too severe for a sample that contains a centerline hole. To verify the extent of centerline deterioration, a second specimen of 0.875-in. diam and 0.850-in. length and without a centerline bore hole was brine-quenched from 830°C. When the specimen was sectioned longitudinally, the microstructure showed massive central voids which had formed during quenching (Fig. 12). The brine-quench cooling rate was evidently too rapid for the U-0.83 wt% Ti alloy at the selected sample diameter.

The remaining portion of the original U-0.83 wt% Ti casting was used for tensile tests to see if ductility was affected by cooling rate. Table IV gives the average tensile-test results for four specimens that were oil- or water-quenched and then aged for 3 h at 381°C. The tensile and yield strengths are similar in both cases; however, ductility values are inconclusive because only two samples were tested for each quenching technique. The hardness values of 420 DPH are slightly higher than values predicted by the aging curves shown in Fig. 10. According to those curves, a hardness of 405 DPH would be expected from an aging time of 3.5 h at 381°C.

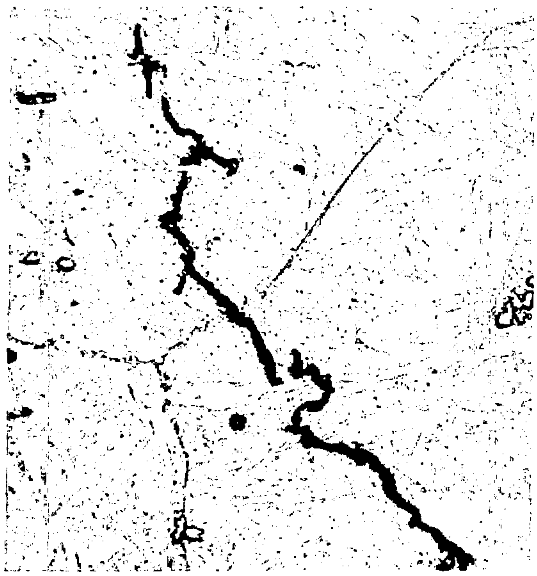
Six tensile-test blanks were machined from a second casting made of U-0.54 wt% Ti and having an impurity level similar to that of the 0.83 wt% alloy. The specimens from this second casting were subjected to the same oil- and water-quenching techniques as were those of the 0.83 wt% alloy. Tensile tests were performed on the as-quenched specimens to see if as-quenched tensile strength and ductility were the same



2.8X

100X

250X



500X

Fig. 11. Center defects in the microstructure of a 10%-brine-quenched specimen with a bore hole.

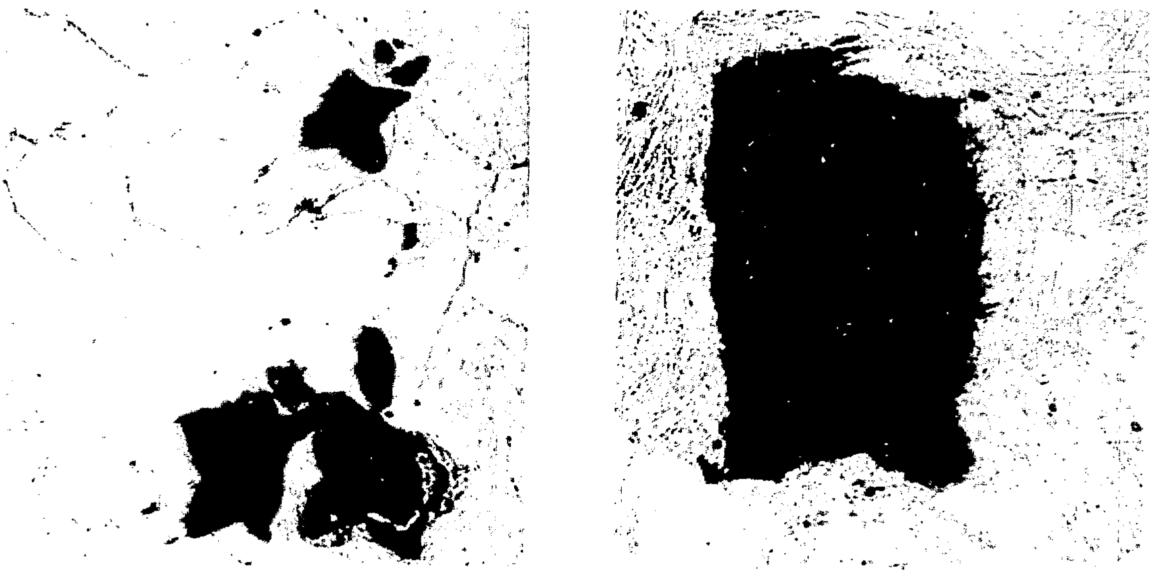


Fig. 12. Center voids in the microstructure of a 10%-brine-quenched specimen without a bore hole. Left, 100X; right, 250X.

for oil- and water-quenched specimens. Table V lists the tensile properties obtained on the as-quenched U-0.54 wt% Ti alloy. Tensile strength and ductility are the same for the different cooling rates in the two quench media.

The butt ends of the tensile-test specimens were examined after testing and the microstructures are shown in Fig. 13. After etching, the metallographic samples were allowed to oxidize in air for 3 days to bring out any differences in grain boundary phase. The microstructures are similar to those of the gamma-quenched specimens shown in Fig. 6b and d.

IV. CONCLUSIONS

As-quenched and aged hardness of U-0.83 wt% Ti alloy is not a function of cooling rate in the range of 38-190°C/sec, despite minor changes in microstructure.

Brine quenching produces centerline defects as a result of the rapid cooling rate, and therefore should not be employed for quenching U-0.83 wt% Ti alloy.

Tensile strength and ductility are not sensitive to quenching rates of 38 and 98°C/sec for U-0.83 wt% and U-0.54 wt% Ti alloys, respectively.

Water and oil quenching should produce the most desirable mechanical properties in quenched and aged dilute uranium-titanium alloys so long as sample geometry produces a center cooling rate of at least 40°C/sec.

TABLE IV

MECHANICAL PROPERTIES OF QUENCHED AND AGED U-0.83 wt% Ti ALLOY

	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%--1 in.)	Hardness (DPH)
Oil-quenched and aged	204 000	146 500	7	420
Water-quenched and aged	205 000	144 700	10	420

TABLE V

MECHANICAL PROPERTIES OF QUENCHED U-0.54 wt% Ti ALLOY

Quench Medium	Tensile Strength (psi)	0.2% Yield Strength (psi)	Elongation (%--1 in.)	Hardness (DPH)
Water	165 400	71 600	11	315
Water	161 400	71 500	10	315
Water	168 000	71 900	13	315
Oil	166 000	73 800	11	325
Oil	169 100	71 600	12	325
Oil	167 100	72 300	12	325

Fig. 13. Microstructure (250X) of quenched and aged U-0.83 wt% Ti alloy tensile-test specimens. Left, oil-quenched; right, water-quenched.