EFFECT OF HUMIDITY ON TENSILE
DUCTILITY OF URANIUM-TITANIUM ALLOYS

by

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ABSTRACT

It was determined that a high relative humidity during tensile testing is detrimental to tensile ductility of the U-0.75 wt.% Ti alloy. For example, longitudinal bars tested in 1% relative humidity showed percent elongations and reduction in areas in excess of 10%; while those tested at 99% R.H. had values less than 5%. Bars tested at intermediate humidities showed intermediate ductility values. The strain rate was 0.005 inches/inch/minute. Only one material condition was investigated: gamma quenched material aged 24 hours at 350°C. Transverse bars were also tested and these showed a corresponding, though smaller, loss in tensile ductility with high relative humidity. The mode of failure was observed and a mechanism is proposed to explain the observed behavior.

INTRODUCTION

Several laboratories, involved in tensile testing of uranium-rich titanium alloys, have observed a larger-than-expected variation in tensile ductility. The variation is observed moreso on a batch-to-batch or day-to-day basis, than on a sample-to-sample basis. The simplest explanation of this behavior is that of a causative variability in chemistry or microstructure (heat treatment) from one batch to the other. Another explanation, although less rational and probable, is that test conditions varied from batch-to-batch or day-to-day causing at least part of the variability. This paper is concerned with one of these test variables, in particular, the relative humidity of the testing environment.

The main reason for considering this variable (i.e., relative humidity) is an observation reported for unalloyed uranium. Whitlow and Williams,⁽¹⁾ and Hughes et al⁽²⁾ observed that unalloyed uranium in certain heat treated conditions showed a reduction in tensile ductility when tested in a 100% relative humidity air atmosphere. The conditions under which this effect occurred were somewhat mystical and elusive, but once found, were somewhat reproducible.

Another reason for looking at the humidity variable is that unalloyed uranium reacts with water vapor to form hydrogen^(3,4,5) as do certain uranium alloys.⁽⁶⁾ It is also known that hydride inclusions are detrimental to tensile ductility in unalloyed uranium.⁽⁷⁻¹⁰⁾ Thus it is

possible that the U-Ti alloys would react during the tensile test with atmospheric water vapor to form hydrogen. This newly generated hydrogen would then enhance crack nucleation and/or propagation thus significantly reducing ductility without appreciable loss in yield or ultimate strengths. This possibility further suggested singling out the humidity variable for study in the U-Ti system.

EXPERIMENTAL

To minimize material variables, all test bars were from the same extruded casting and all bars received the same thermal and mechanical treatments. More specifically, a previously arc-cast U-0.75 wt% Ti ingot was induction cast into a hollow cylinder. The hollow cylinder was alpha extruded (salt bath at 625°C) to an 8 inch diameter tube with a 1 inch wall thickness. Three rings, each 3-1/2 inches long, were cut from the tube and vacuum outgassed 5 hours at 525°C, at 5×10^{-6} mm of Hg⁰. The rings were then solutionized 20 minutes at 800°C in an argon atmosphere furnace and water quenched to ambient. The rings were aged 24 hours at 350°C in a vacuum furnace at 5×10^{-6} mm of Hg⁰ pressure and helium gas quenched to room temperature. This sequence of thermal and mechanical treatment yields a material having optimum toughness. Radiographing of thin sections, both longitudinal and transverse, of the rings failed to show any evidence of microcracking in the heat treated rings.

Twenty tensile bars transverse to the extrusion direction and 20 longitudinal bars were machined from each of the three rings. A round tensile test bar was used. The dimensions were 3 inches long by 3/8 inch diameter. The reduced section was 1.25 inches long with a diameter of 0.252 ± 0.005 inches. A 0.18 inch radius fillet connected the reduced section to the grip area. The gage length was 1.00 ± 0.005 inch. The grip area was 5/8 inch long with 16 threads per inch. The bars were pulled at a strain rate of 0.005 in./in./min.

Immediately prior to testing, the tensile test bars were cleaned by: a) electrocleaning for 4 seconds in an orthophosphoric acid bath to remove the dark oxide surface film, followed by ultrasonically cleaning in ethyl alcohol; or, b) cleaning ultrasonically in ethyl alcohol alone (sample therefore tested with dark oxide film). It is significant that samples tested with or without the dark oxide film (UO_2) both showed the same response to testing in humid and dry environments.

The tensile test bars were tested at several conditions of wetness and dryness; a) 0% RH (This was achieved by a mechanical and oil-diffusion pump producing a vacuum environment at a pressure of 3×10^{-5} mm of Hg^0 .); b) 1% RH (Achieved by the desicating action of 1/2 liter of silica gel in a 10 liter test chamber for 4 hours. A humidity probe showed the average relative humidity after 4 hours equilibration to be 0.7 percent.); c) 10% RH (These tests were conducted in a room carefully controlled to a 10% RH level.); d) 50% RH (These tests were conducted in a room controlled to approximately $50\% \pm 10\%$.); e) 99% RH (Achieved by equilibrating 4 hours in a 10 liter test chamber containing 1 liter of distilled water. Since no condensed water was ever observed on the walls of the container or the sample, the value of 99% RH was assumed.); and 100% RH (Achieved by total immersion of the sample in distilled water.)

RESULTS

Tests in Various Environments

The results of tensile tests for bars longitudinal to the working direction under known conditions of wetness and dryness are shown in Figure 1. The results for transverse bars under similar conditions are shown in Figure 2. The following conclusions are drawn from these data:

- 1) For the one mechanical and thermal treatment tested, the degree of wetness or dryness during a standard tensile test does influence the measured tensile ductility (i.e., percent elongation and reduction in area).
- 2) A high humidity reduces measured tensile ductility values. A low humidity improves tensile ductility.
- 3) To a first approximation, the change in tensile ductility is linearly proportional (inversely) to humidity. Keep in mind, however, that there is not enough data to justify the dashed straight lines drawn in Figures 1 and 2.
- 4) Both longitudinal and transverse bars exhibit a loss in tensile ductility; however the effect is more significant for longitudinal bars. This indicates an anisotropy effect.
- 5) Transverse bars, at low and medium humidities, are less ductile in a tensile test than are longitudinal bars. At high humidities, the transverse bars are

more ductile.

6) Longitudinal bars, at low humidity levels, show a much larger difference between % reduction in area and % elongation than the transverse bars do. At medium and high humidity levels there is little difference between the two measures of ductility.

7) The transverse bars show slightly higher yield strengths than do the longitudinal bars at medium and low humidity levels.

8) The transverse and longitudinal bars have approximately the same ultimate tensile strengths for a given humidity level in the low and medium humidity range.

9) The change of yield and ultimate tensile strength with humidity is very small at low and medium levels. At high humidities, there is an apparent increase in yield strength and decrease in ultimate tensile strength.

Chemical Purity of Test Bars

Several test bars were chemically analyzed after pulling in laboratory air. One transverse and one longitudinal bar from each of the three starting rings were analyzed. The average of these six analyses are shown in Table 1. The upper and lower limits, when shown, do not represent the accuracy of the test, but merely the range of the six chemical analyses. The low levels of hydrogen (0.80 ± 0.30 ppm) and carbon (25 ± 10 ppm) as well as the constancy of titanium

content are worthy of note (0.80 ± 0.02 wt.%). One can conclude from Table 1 that the alloys are of high purity and that they are homogeneous from bar to bar.

DISCUSSION

Uranium-Water Reaction

The uranium-water and uranium-water-oxygen reactions have been studied in the absence of stress.⁽³⁻⁵⁾ The reaction of uranium with water vapor ($U + 2H_2O \rightarrow UO_2 + 2H_2$) is dependent on the partial pressure of water⁽⁴⁾ as is the amount of hydrogen evolved from the reaction. Baker et al⁽⁴⁾ found the hydrogen generated was from 1 to 13% less than theoretical with the greatest deficiencies occurring at 100% relative humidity. The hydrogen which is not evolved may enter the uranium and cause the reduction of ductility observed in uranium specimens tested in wet air.⁽¹⁾

Hydrogen Embrittlement in Uranium

The effect of hydrogen content on ductility in uranium was studied by several investigators.⁽⁷⁻¹⁰⁾ Marsh et al⁽⁷⁾ reported that the ductile-brittle transition temperature was raised 20°C by the addition of 4.7 ppm hydrogen to alpha annealed uranium. Davis⁽⁸⁾ investigated the tensile behavior of uranium-hydrogen alloys containing 0.1 to 2.0 ppm hydrogen over the temperature range 20°C to 100°C. These alloys exhibited a marked decrease in ductility at hydrogen contents of 0.2 to 0.4 ppm hydrogen. Above 0.4 ppm hydrogen the elongation to fracture remained constant and independent of hydrogen content. Davis⁽⁸⁾ interpreted his results in the following way: 0-0.2 ppm hydrogen was absorbed by uranium carbide and other precipitates; 0.2-0.4 ppm hydrogen was

taken into solution by the uranium metal; and between 0.4-2.0 ppm uranium hydride precipitates were formed. He concluded that hydrogen in solution was the primary source of embrittlement. Gardner and Riches⁽¹¹⁾ reported the presence of uranium-hydride precipitates in uranium containing as little as 0.12 ppm hydrogen. At these low hydrogen concentrations the hydride phase formed predominantly as a grain boundary network but increasing hydrogen contents resulted in the formation of a more discrete and widely separated grain boundary precipitate. They also studied the effect of uranium hydride on the tensile properties of uranium and reported that reductions in elongation were accompanied by an increase in the yield strength of β -quenched uranium-hydrogen alloys. They concluded that the increase in yield strength was due to the formation of fine uranium hydride precipitates.

Beevers and Newman⁽¹⁰⁾ studied the metallographic and mechanical behavior of uranium thermally charged to contain between 1.23 and 15 ppm hydrogen. They concluded that the embrittlement occurred primarily through the formation of uranium hydride precipitates rather than a matrix solid solution cause. The degree of embrittlement was related to the size of the hydride precipitates with maximum length of hydride particle and maximum embrittlement occurring at roughly 2.5 ppm hydrogen in thermally charged alloys. With hydrogen contents greater than 2.5 ppm, there was a continuous decrease in maximum length of the hydride particles and an

accompanying increase in fracture stress. Beever and Newman⁽¹⁰⁾ also determined that uranium, like α -titanium and α -zirconium is an exothermic absorber of hydrogen. This was partially established by the observation that embrittlement was enhanced by increased strain rate. Endothermic absorbers such as the body centered cubic metals, show increasing embrittlement with increasing strain rate. They also show increasing embrittlement with increasing hydrogen content, and the embrittlement is primarily due to the presence of hydrogen in solution. In the case of uranium, the fracture stress decreased starting from zero hydrogen content reaching a minimum at 2.5 ppm hydrogen and increased thereafter in the range studied. It is suggested by Beever and Newman⁽¹¹⁾ that the embrittlement in uranium is due to a low fracture stress of the inclusions and that these cracked inclusions introduce cracks into the metal matrix.

Humidity Versus Tensile Ductility in Uranium

Whitlow and Williams⁽¹⁾ according to Hughes et al.⁽²⁾ first reported the effect of humidity on tensile ductility of unalloyed uranium. They⁽²⁾ observed the effect for both beta-quenched and warm-rolled unalloyed uranium when testing at ambient temperature in moist air (~100% R.H.). During their tests, low ductility values were obtained in the very moist air, but the UTS was only marginally reduced. Hydrogen embrittlement and stress corrosion cracking were suggested as possible mechanisms. An interesting aspect of this work was that the effect of high humidity was shown to be independent

of the carrier gas. Others^(4,5) have shown that in the absence of stress, oxygen exerts an inhibiting effect on the water-uranium reaction. Furthermore, it has been shown⁽⁵⁾ that in the presence of oxygen no hydrogen is evolved as a result of the uranium-water reaction.

To digress for a paragraph and to expand on the last two sentences; one of the more interesting aspects of the oxidation of uranium in humid environments is that the overall oxidation is retarded when O_2 is present.^(3,5,12) A reduction of the rate by as much as two orders of magnitude was observed by Baker et al.^(4,5) They showed using oxygen isotopes that while water is the primary oxidizer, there is no net consumption of the water until the oxygen is consumed. The majority of hydrogen liberated from the uranium-water reaction combines with the oxygen, which is probably absorbed on the surface, to form more water. Their data also showed when both oxygen and water are present, that there is little dependence of the reaction rate on oxygen or water partial pressures.

Returning to the question of relative humidity versus tensile ductility for uranium metal, Hughes et al.⁽²⁾ further investigated and confirmed the effects reported by Whitlow and Williams.⁽¹⁾ It was established by Hughes et al.⁽²⁾ that a combination of certain factors is required without any one of which the detrimental effect of moisture (~100% R.H.) at ambient temperatures is no longer manifest. These factors are:

1) Specific surface oxidation resulting in the U_3O_8 type.

(Acid cleaned specimens did not exhibit ductility loss in high humidity environments, but did so when oxidized under appropriate conditions to form U_3O_8 .)

2) Particular metallurgical conditions. (The effect was confined to warm-worked and gamma-quenched material.

Six other metallurgical conditions tested did not show a loss in ductility in very moist air.)

3) Specimen shape. (Rectangular sectioned specimens showed the effect; circular sectioned specimens did not.)

They postulated that crack initiation, rather than propagation, was critical to the embrittlement effect. The absence of subsidiary cracking in fractured specimens they observed supposedly supported this contention.

At a test temperature of $0^{\circ}C$ (condensed water visible on sample), they⁽²⁾ observed that the above factors were no longer necessary for a loss in ductility. This observation led them⁽²⁾ to conclude that the presence of condensed water (c.g., at $0^{\circ}C$) on the metal surface is far more effective in reducing ductility than a high humidity environment. Consequently the requirement of surface oxide and metallurgical condition, prerequisites for a humidity effect were no longer required when condensed water was present.

MICRO AND MACROSTRUCTURE PRIOR TO FRACTURE

The prior-gamma grain size of the alloy was ASTM 3-4. The hardness was 440 DPN at a 500 gram load. The microstructure was essentially 100% single phase alpha prime in a martensitically-formed acicular structure. Undoubtedly much titanium has clustered into pre-precipitates (G.P. zones) of U_2Ti , but since no distinct noncoherent precipitates have formed, the structure is still referred to as supersaturated alpha, i.e., alpha prime. The inclusions observed were UO_2 and TiC and were somewhat aligned along the working direction. No hydrides were observed. Likewise no microcracking was observed.

The principle macroscopic feature was macrobanding aligned parallel to the direction of working, i.e., the longitudinal direction. The macrobanding arises during alloy solidification and consists of bands of titanium segregation that form parallel to the freezing interface. The interband spacings are of the order of 1/16 inch.

HUMIDITY VERSUS TENSILE DUCTILITY IN U-Ti ALLOYS

The shape of the stress-strain curves for samples tested in wet and dry conditions were approximately identical except for the length of the test. This is apparent in Figures 1 and 2, which showed the loss in tensile ductility was not accompanied by a significant change in yield or ultimate strength. The high ductility values (low humidity) were obtained either through non-initiation of cracks until the later stages of

the test or by slow crack propagation rates as compared to the rate of application of stress. In the study, the presence of high humidity has been shown to have a deleterious effect on tensile ductility. This can be interpreted in terms of increased crack propagation rates due to either chemical action, or lowering of surface energy by a condensed water phase within the crack tip.

The possibility that cracks existed in the bars prior to testing was of major concern after the detrimental effect of humidity was observed. However, as mentioned earlier, the starting material was radiographed and showed no internal or surface flaws. To check for smaller internal and surface flaws, unstrained grip and fillet areas were sectioned and examined at high magnification (100-800X) in the as-polished and lightly etched condition. No internal cracks or seams were observed, and no surface flaws with significant depth were observed. The surface flaws that were observed were few and probably the result of inclusion removal during machining. It is concluded that the bars were essentially crack free prior to testing.

The question of whether atmospheric water vapor promotes crack initiation now arises. The ductility-humidity effect was noticed on electrocleaned samples (no dark oxide) and on samples having the dark oxide. Thus, an oxide-rupture mechanism causing crack initiation in the alloy matrix was discarded. In post-mortem microscopic examination of the reduced section, no evidence was found to support the belief

that humidity was instrumental in crack nucleation.

One observation on microscopic examination of reduced sections was the appearance of microvoids (formed during deformation) which were often associated with inclusions. During deformation, the inclusions fracture (due to their brittle nature) and introduce cracks into the matrix alloy. These matrix cracks under the tensile load stress enlarge into microvoids. In the interior of the tensile bars, the microvoids coalesce to form the fracture zone. A microvoid intersecting the surface of the tensile bar (circumferential cracking) would provide a route for atmospheric water vapor to enter the picture.

The freshly exposed metal at the circumferential crack undergoes a water uranium reaction ($U + 2H_2O \rightarrow UO_2 + 2H_2$ or $U + \frac{8}{3} H_2O \rightarrow \frac{1}{3} U_3O_8 + \frac{8}{3} H_2$). The available oxygen reacts with part of the generated hydrogen to form more water ($H_2 + \frac{1}{2} O_2 \rightarrow H_2O$). Part of the hydrogen reacts with metal where the tensile stress is greatest (i.e., the crack tip) forming a sub-hydride or hydride ($U + \frac{3}{2} H_2 \rightarrow UH_3$). The larger volume occupied by the hydride or sub-hydride creates additional tensile stress along the sample axis and adds to the tensile load stress. This causes rupture in the newly formed sub-hydride exposing fresh metal to react with the existing condensed water in the crack generating additional hydrogen. If the capillary crack contains only condensed moisture (i.e., no atmospheric oxygen) then the newly generated hydrogen is all available to form sub-hydride at the point of

maximum tensile stress resulting in further rupture of the hydride and the exposure of fresh metal to react with available moisture. This process repeats itself resulting in an accelerated crack propagation and an attendant loss in tensile ductility. At the lower levels of humidity, less moisture is available at the crack tip, thus decreasing the rates of the various reaction, and, hence, improved tensile ductility.

One could build a similar argument around the lowering of surface energy by a condensed water phase within the crack tip. However, experimental observation does support the hydride or sub-hydride wedge mechanism. For example, contrast Figures 3 and 4. Figure 3 is a photomicrograph near the surface of a bar pulled in a 1% R.H. air atmosphere. Numerous circumferential cracks are visible (due to the length of test, i.e., 17% elongation and 28% reduction-in-area) and there is no visible hydride directly associated with these cracks. Figure 4 is a similar view of a sample pulled in 99% R.H. air atmosphere. No circumferential cracks are visible (due to short time of test, i.e., 3% elongation and 3% reduction-in-area) and numerous hydride-like platelets are visible near the surface of the bar. This indicates that atmospheric moisture does indeed react with the strained sample to form hydrogen and that this hydrogen is readily absorbed into the alloy to form a second-phase hydride which is probably UH_3 . Figure 5 shows UH_3 in a subsidiary crack along the fracture interface of bar tested in a 99% R.H. air atmosphere. However,

it should also be pointed out that many subsidiary cracks observed in wet-pulled bars did not have visible hydride associated with them.

SUMMARY

Relative humidity is detrimental to conventional strain-rate tensile ductility in the U-0.75 wt. % Ti alloy heat treated to maximum toughness. The loss in tensile ductility is proportional to the increase in percent relative humidity. The effect occurs for samples taken both longitudinal and transverse to the working direction although the effect is not as strong for transverse bars. The observed loss in ductility is probably the result of an alloy-moisture reaction which generates hydrogen. Part of this hydrogen is available for further reaction with the alloy. When circumferential cracks, which form naturally during the course of the deformation process, intersect the surface; the alloy-moisture generated hydrogen forms a hydride or sub-hydride at the crack tip. The increased volume at the crack tip enhances crack propagation, which causes the observed loss in tensile ductility.

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TABLE 1. Chemical analyses of six representative tensile bars (one longitudinal and one transverse bars from each of the three starting rings). The chemical analyses were on tested (broken) tensile bars pulled in laboratory air.

Al	12 ± 5*ppm	K	<10	V	<1
B	<1	Li	<1	Zn	<10
Ba	<1	Mg	<1		
Be	<1	Mn	5		
Bi	<1	Mo	8±5	Ti	0.80±0.02 wt.%
Ca	<1	Na	<1	H	0.80±0.30 ppm
Cd	1	Nb	<10		
Co	<1	Pb	5	C	25±10 ppm
Cr	10	Sb	<1	N	20±15
Cu	10	Si	38±5		
Fe	45±20	Sn	<5		

*The upper and lower limits, when shown, do not represent the accuracy of the test, but merely the range of the six chemical analyses.

LIST OF FIGURE CAPTIONS

Figure 1. Tensile test values for longitudinal bars as a function of the relative humidity during testing. The number of bars tested per datum point is in parentheses. The range of measured values is given by the vertical lines intersecting the averaged datum points.

Figure 2. Tensile test values for transverse bars as a function of the relative humidity during testing. The number of bars tested per datum point is in parentheses. The range of measured values is given by the vertical lines intersecting the averaged datum points.

Figure 3. Photomicrograph of side of tensile bar tested in one percent relative humidity at room temperature. Numerous circumferential cracks are visible (due to length of test, i.e., 17% elongation and 28% reduction in area), and there is no visible hydride directly associated with these cracks. X200.

Figure 4. Photomicrograph of side of tensile bar tested in 99 percent relative humidity at room temperature. No circumferential cracks are visible (due to short time of test, i.e., 3% elongation and 3% reduction in area), and numerous hydride platelets are visible near the surface of the bar. X400.

Figure 5. Fracture zone of the test bar shown in Figure 4 (i.e., tested in 99% relative humidity). Hydride is associated with a subsidiary crack along the fracture interface. X400.

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