TENSILE PROPERTIES OF TITANIUM ELECTROLYTICALLY CHARGED WITH HYDROGEN

by Robert J. Smith and Dumas A. Otterson

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Yield strength, ultimate tensile strength, and elongation were studied for annealed titanium electrolytically charged with hydrogen. The hydrogen was present as a surface hydride layer. These tensile properties were generally lower for uncharged titanium than for titanium with a continuous surface hydride; they were greater for uncharged titanium than for titanium with an assumed discontinuous surface hydride. We suggest that the interface between titanium and titanium hydride is weak. And the hydride does not necessarily impair strength and ductility of annealed titanium. The possibility that oxygen and/or nitrogen can embrittle titanium hydride is discussed.
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SUMMARY

The yield strength, ultimate tensile strength, and elongation were studied for annealed titanium electrolytically charged with hydrogen. The hydrogen was present as a hydride on the titanium wire surface as islands or a completed layer or sheath depending on the total hydrogen content. The tensile properties were greater for uncharged titanium than for titanium with an assumed discontinuous surface hydride (islands); they were generally lower for uncharged titanium than for titanium with a continuous surface hydride. It follows that hydride formation does not necessarily impair the strength or ductility of annealed titanium. The interface between titanium and titanium hydride is weak. The idea that oxygen and nitrogen impurities may help cause the hydride to be brittle is also presented. The possibility that the hydride sheath may provide a barrier against corrosion and further hydrogen contamination is discussed.

INTRODUCTION

Hydrogen embrittlement of titanium (Ti) is ascribed to the presence of the hydride phase. Either impact or low-strain-rate embrittlement may occur depending upon when and how the hydride is formed (refs. 1 to 3). Two of the existing explanations for hydrogen embrittlement seem contradictory. One states that the hydride itself may be brittle (refs. 1 and 4). The other states that the hydride possesses some plasticity and that cracks start in the weak interfaces between the titanium (hexagonal close packed) and the hydride (face-centered cubic) (ref. 2). These statements may be rationalized if we consider that the presence of other impurities and/or the actual hydrogen content of the hydride may be involved in its ductility or brittleness. Longson has shown that hydrogen embrittlement of niobium (Nb) is enhanced by an increase of the oxygen and nitrogen content (ref. 5). Sasaki and Amano have shown that no detectable amount
(20 ppm) of oxygen and nitrogen is added during the electrolytic preparation of niobium hydride even though contamination by these impurities can hardly be avoided during the absorption of hydrogen gas above 473 K (200°C) (ref. 6). Because Ti also reacts readily with oxygen and nitrogen at elevated temperatures, electrolytic charging of Ti with hydrogen should have less oxygen and nitrogen pickup as compared to absorption from hydrogen gas at elevated temperature.

In this work, we study some of the mechanical properties of titanium hydride. The properties are the yield strength (YS), ultimate tensile strength (UTS), and uniform elongation. We charged the Ti with hydrogen electrolytically. By using this technique, we were able to prepare specimens with approximately the desired hydrogen contents and to minimize the oxygen and nitrogen contamination. Also, this type of hydrogen absorption is somewhat similar to that due to corrosion (ref. 7). This work is part of a program to determine the mechanism of hydrogen embrittlement in structural metals.

EXPERIMENT

Specimen Preparation

Treatment of specimen prior to charging. - The specimens were titanium wires of 99.96-atomic-percent purity. This was confirmed by spectrographic analysis at this laboratory. Table I presents the concentration of 34 impurities as reported by the manufacturer. The specimens were 8 centimeters long and 0.254 millimeter in diameter.

All the specimens were individually annealed by joule heating to approximately 1075±25 K for 2 to 2.5 hours in a vacuum. The vacuum was not permitted to be degraded beyond 10⁻⁷ torr (~10⁻⁵ N/m²) by the gases liberated when the specimen temperature was increased to 1075 K. Both oxygen and nitrogen were found by mass spectrometry in the gases liberated below 770 K (500°C). But no oxygen was found if the liberated gas was allowed to remain in the vicinity of the specimen until the system reached temperatures much above 770 K. These observations indicate that some oxygen and nitrogen were removed from the specimen when its temperature was increased slowly up to the annealing temperature with pumping. However, a major analytical effort would be required to evaluate the amount of these gases removed. And this is beyond the scope of the present study. During the annealing at 1075 K and subsequent cooling, the vacuum was 2×10⁻⁸ torr (~3×10⁻⁶ N/m²). The specimen was cooled slowly by reducing the joule current to zero in approximately 30 minutes.

The annealed wires were rinsed in acetone and then chloroform to remove any grease due to handling. The wires were lightly etched in a 1:1 hydrofluoric acid solution for about 10 seconds, then rinsed thoroughly with distilled water, and finally stored in a
1 molar phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) solution. This procedure permitted the specimen to be charged with hydrogen in a more consistent manner.

**Electrolytic charging with hydrogen.** - The entire lengths of the Ti tensile specimens were charged with hydrogen. The electrolysis cell was merely a container with the specimen as the cathode, a platinum ribbon as the anode, and 1 molar H\textsubscript{3}PO\textsubscript{4} as the electrolyte. We found that small amounts of freshly precipitated hydrated Ti oxides would dissolve in this solution. Fresh electrolyte was used with each specimen. The current density was about 8 milliamperes per square centimeter for most of the runs and 40 milliamperes per square centimeter for the remainder. The charging periods ranged from about 6 minutes to 118 hours. This gave us bulk hydrogen-titanium atom ratios (expressed as H/Ti) from 0.002 to 0.33.

**Tensile Tests**

Preliminary tests on annealed Ti specimens and on specimens charged electrolytically to bulk H/Ti values of 0.02, 0.23, and 0.29 indicated that tensile properties were strain-rate sensitive. The lowest strain rates gave the lowest values for YS, UTS, and elongation. All specimens reported in this investigation were pulled in tension in air by an Instron tensile testing machine at its slowest crosshead speed of 0.051 millimeter per minute. The specimen was held by two steel grips. One grip was attached to the load cell and the other to the movable crosshead of the tensile machine. The wire was wrapped once around a 4.4-millimeter-diameter post on each grip and secured to the grip by being tightened at the end under a bolt. The length of the test section (length of wire between post centers) was 25.4 millimeters. The data were obtained in the form of load-elongation curves on strip-chart recordings. The 0.2 percent YS, UTS, uniform elongation, maximum elongation, and fracture stress were obtained from these charts. The elongation of a number of specimens was also measured with a cathetometer and by sequential photography. These three techniques yielded consistent results.

**Specimen Characterization**

**Determination of hydrogen in titanium.** - A method was developed to determine the hydrogen content in Ti wire specimens which was rapid, accurate, and sensitive. It was a modification of one used in a similar study of the palladium-hydrogen system (ref. 8). This method involved the desorption of hydrogen gas and its determination in a mass spectrometer capable of 1 percent accuracy.

This method can be used to determine hydrogen in Ti only if the hydrogen can be desorbed quantitatively. Hydrogen solubility and diffusion calculations based on the work of Albrecht and Mallett (ref. 9) indicated that all but a trace of hydrogen could be re-
moved from Ti at 1070 K (800°C). These calculations indicated that the H/Ti for the 0.25-millimeter-diameter specimens would be near 0.0005 after 1/4 hour at 1070 K if the final hydrogen pressure was about 5×10^{-4} torr (~0.07 N/m²). With a two-step desorption process at 1070 K, the final hydrogen pressure was rarely more than 5×10^{-4} torr (~0.07 N/m²). This desorption would be quantitative for most of our specimens under these conditions. It should be noted that the amount of hydrogen desorbed in a third desorption step was generally negligible.

At 1070 K, Ti reacts with water to produce hydrogen. Because water can desorb from the quartz walls of the vacuum system when it is heated to 1070 K (800°C), it was necessary to establish the conditions which yielded acceptably low and consistent blanks. The blanks were determinations carried out with desorbed Ti specimens. Blank H/Ti values of 0.004±0.002 were obtained with a two-step desorption at 1070 K by adhering to the following conditions:

1. Heating as little of the quartz as possible
2. Heating the same amount of quartz during each analysis
3. Heating the evacuated system to 1070 K daily prior to doing any analysis
4. Heating the system to 570 K (300°C) while evacuating after adding the specimen
5. Preventing direct contact between Ti and quartz by use of a closed-end gold tube

The uncertainty in the blanks indicates that the error in the analytical method for H/Ti would be near ±0.002. The error would only be appreciable for specimens with lower H/Ti values. Because of this error, the hydrogen content of annealed specimens was estimated using the solubility relation of Albrecht and Mallett (ref. 9). For the annealing conditions (1075 K and 2×10^{-8} torr), the H/Ti value was estimated to be 0.00005. Even though improved accuracy is needed before a detailed study of tensile properties of slightly charged Ti is made, the results were sufficiently accurate to compare its tensile properties with those of uncharged Ti and with more heavily charged Ti.

The bulk H/Ti was determined for the two portions of the specimen immediately adjacent to the point of rupture. Uniformity of charging was spot checked by analyzing samples taken from various positions along specimen length. Bulk ratios are specified because the hydrogen is concentrated near the surface. In most instances, the variation in results was close to that reported for blanks. This was true for all specimens with H/Ti < 0.01. The data were discarded for those specimens where the difference in bulk H/Ti was 0.01 or more. We believed such large error indicated that the hydrogen was unevenly distributed along the length of the specimen.

Investigation of radial distribution of hydrogen. - Radial distribution of hydrogen was investigated by three different techniques. First, photomicrographs were made of cross sections of selected Ti specimens to check for the presence and continuity of a surface layer.

Then, Laue patterns were used to identify the phases present at or near the speci-
men surfaces. Laue patterns were also made with specimens which had previously been etched to remove the hydride layer.

Finally, hydrogen determinations were made on etched and unetched specimens to learn if the hydrogen was located near the surface of the charged specimens. They were etched 3 minutes in a mixture that was 1 part hydrofluoric acid and 10 parts nitric acid.

RESULTS

Hydrogen Distribution

Results obtained from the photomicrographs, Laue diffraction patterns, and comparison of hydrogen contents of etched and unetched specimens lead to the same conclusion. They indicate that a surface layer or sheath was indeed formed by electrolytic charging of Ti with hydrogen.

Comparison of photomicrographs in figure 1 shows the presence of a surface layer or sheath which varies in thickness with H/Ti. The sheath is virtually continuous for H/Ti as low as 0.02. The thickness depends on the charging time. However, the rate of increase in thickness decreases with increasing charging time and approaches zero for specimens charged long enough to reach H/Ti near 0.4. The relation between sheath thickness and hydrogen content was found to be roughly linear. The sheath is discernable on all specimens except for H/Ti $\approx$ 0.005. In this case, we assume the surface layer is either too thin to be observed or is discontinuous and missing from the area photographed.

Williams, Koehl, and Bartlett have shown that isolated spots and thin discontinuous layers of hydride are formed when hydrogen gas initially reacts with specially clean Ti surfaces at room temperature (ref. 10). One may assume that electrolytic absorption at room temperature proceeds in like manner.

Laue patterns support this sheath description. Only the $\gamma$-phase was observed for H/Ti above 0.125. Both $\alpha$- and $\gamma$-Ti lines appeared for H/Ti between 0.009 and 0.125. Only $\alpha$-Ti was found on specimens with H/Ti equal to 0.009 and on hydrided specimens which had been etched to remove a 0.025-millimeter-thick layer from the surface. Because copper K-\(\alpha\) X-rays penetrate 4 to 5 microns into $\gamma$-Ti, the $\alpha$-Ti lines can appear in some Laue patterns even though the hydride sheath is complete.

Comparison of the hydrogen content of etched and unetched portions of specimens indicates that virtually all the hydride can be etched off from hydrided specimens. H/Ti values of 0.007$\pm$0.003 were found after etching specimens that had been charged to H/Ti $\approx$ 0.3. After etching annealed Ti (H/Ti $\approx$ 0.004$\pm$0.002), H/Ti values of 0.006$\pm$0.002 were obtained. It is evident that the H/Ti value for the core is less than 0.007$\pm$0.003 and is probably closer to 0.001 (20 ppm). This value is obtained by subtracting
the hydrogen content of etched annealed Ti. In this way, corrections are made for the hydrogen added to the core during etching as well as for the normal blank. The hydrogen solubility in Ti at room temperature has been estimated to be as low as 1 ppm (ref. 1) and as high as 20 ppm (ref. 11). Hence, these results indicate that virtually none of the hydrogen added by electrolytic charging is found in the core of the specimen.

The very low hydrogen content of the core permits the estimation of the hydrogen content of the sheath by using photomicrographs to obtain its thickness. The H/Ti values of the surface layer calculated in this way ranged from 1.0 to 1.2. The highest value was obtained when we corrected for irregularities in the sheath. There is obviously error in these values, but they do imply that the H/Ti of the sheath is near the lowest reported value for the γ-phase. Libowitz (ref. 12) reports that the γ-phase can have H/Ti values which range from 0.9 to 1.99.

**Tensile Data**

The YS, UTS, and uniform elongation are shown as functions of bulk H/Ti in figure 2. Minimums in these properties seem to occur near H/Ti = 0.01. For H/Ti > 0.02 these tensile property values are generally greater than those values for annealed uncharged Ti. This is not as evident for the elongation because of the greater scatter in the data. However, the average increase in the elongation for H/Ti > 0.02 is 7.87 percent, while the average increase for annealed uncharged Ti is 7.18 percent when all the data points are used. In table II are listed the values for YS, UTS, FS (fracture stress), uniform elongation, maximum elongation, the charging data, and bulk H/Ti. It should be stated that the relatively low elongation of our annealed Ti is due to the annealing temperature and not to the impurity content of the specimen. We have evidence that shows that the elongation would have been greater if a lower annealing temperature had been used.

**DISCUSSION**

Our results showed that composite specimens made up of a surface hydride surrounding a Ti core were obtained as the result of electrolytic charging of Ti with hydrogen. We shall discuss the barrier effects of this sheath and the effect of such a sheath on the tensile properties of the specimen. In so doing, we shall be able to infer some of the tensile properties of Ti hydride.
Barrier Effect of Hydride Sheath

The hydride sheath can act as a barrier to hydrogen diffusion as well as to chemical or corrosive attack.

The barrier to hydrogen diffusion is indicated by the decrease in growth rate of the sheath as electrolysis continues. The decrease in growth rate was reported earlier by others who interpreted it as meaning that the hydrogen diffusion rate is less in the hydride than in Ti (ref. 10). The result could, of course, be interpreted as due solely to the increase in the \( \gamma \)-phase thickness. Any hydrogen which permeates the sheath does not increase the hydrogen content of the \( \alpha \)-Ti above its equilibrium value but forms more hydride immediately adjacent to the sheath. The hydride will precipitate from \( \alpha \)-Ti containing as little hydrogen as a few tens per million parts (i.e., \( H/Ti > 0.001 \), ref. 1).

Regardless of explanation, however, the decrease in growth rate of the sheath does indicate that the \( \gamma \)-phase can act as a barrier to hydrogen diffusion.

The evidence which indicates that the hydride layer might act as a barrier to chemical or corrosive attack is provided by Otsuka (ref. 13). He found that titanium hydride formed by electrolytic charging has chemical properties different from those of \( \alpha \)-Ti. The hydride is insoluble in 24 percent hydrochloric acid, while the \( \alpha \)-Ti dissolves until a continuous hydride layer is formed on its surface. The possibility exists that the hydride is less soluble than Ti in other corrosive agents; that is, the hydride can act as a protective coating if present as a continuous layer. This quality of the hydride would not be observed where the hydride is dispersed as particles in a Ti matrix.

Role of Hydride Sheath on Tensile Properties

Hydrogen embrittlement of Ti, as mentioned earlier, has been ascribed to the presence of the hydride phase. However, our investigation shows that the presence of the hydride phase may enhance as well as degrade the YS, UTS, and elongation. Lenning, Craighead, and Jaffee (ref. 3) reported that hydrogen increased YS and UTS but that hydrogen decreased the elongation and ductility. The hydrogen was distributed uniformly throughout their specimens; in ours, virtually all of the hydrogen was located near the surface as a hydride layer or sheath.

The ductility of our specimens is evident from our data and in the crude evidence that all the wires could easily be wrapped around a 4.4-millimeter mandrel for tensile testing. The data (table II) show that the maximum elongation exceeds uniform elongation in all cases. Also, the FS based on the original cross section is generally about 0.7 UTS. These results indicate some reduction in area, which is the more usual criterion for ductility. Necking (i.e., reduction in area) was also observed visually.

We have already noted that the hydride layer or sheath is virtually complete when the bulk \( H/Ti \approx 0.02 \). At this concentration, the tensile properties are at or near their
maximum values. However, the minimum values for tensile properties occur when H/Ti is between 0.005 and 0.01. In this concentration range, we assumed the hydride to be present on the Ti surface as isolated spots as reported by Williams, Koehl, and Bartlett (ref. 10). An obvious difference between the two types of hydride coverage is in the geometry of the interface between the two phases. When the layer is continuous, the interface is parallel to the applied tensile stress. When the layer is discontinuous in the form of spots or islands, portions of the interface are on the surface. Hence, some parts of the interface will lie transverse to the applied tensile stress.

Effect of Isolated Islands of Hydride

For specimens of H/Ti in the neighborhood of 0.01, where we assume only hydride spots or islands on the surface, the tensile properties of the hydride are of little importance. The interfaces between the hydride (γ-Ti) and the α-Ti which lie perpendicular to the direction of tension become the regions of concern. If we assume that the cohesive energy across the interface is lower than that of the hydride or α-Ti, then the fracture would start at the interface. The thickness of the hydride (a function of total hydrogen content) would vary the cross section of the α-Ti in this region and the size of this initial crack.

Possible Tensile Properties of Hydride Sheath

Let us consider two alternatives for the tensile properties of the hydride sheath and their possible effects on the tensile properties (YS, UTS, and elongation) of the specimens with a continuous sheath. First, the tensile properties of the hydride may be much lower than those of α-Ti, or second, the tensile properties may be near or greater than those of α-Ti.

If the tensile properties of the hydride are low, then the enhancement observed must be explained by increased tensile properties of the Ti core. One explanation is that the surface layer or film reduces the probability that surface defects cause failure. However, once the hydride film has formed, additional hydride has little effect on this probability and only tends to decrease the cross section of the Ti. This decrease in cross section now requires that the tensile properties of the α-Ti increase in order to get the reported results. For bulk H/Ti values of 0.33 the cross section of Ti is only 70 percent of its precharged cross section. If the strength of the sheath is negligible, then the YS and UTS of α-Ti must increase by at least 50 percent to account for the measured values. Lenning, Craighead, and Jaffee (ref. 3) report that evenly distributed hydrogen can produce such large increases in YS and UTS. About 4 atom percent of hydrogen is needed to produce a 50-percent increase in YS, and about 21 atom percent
of hydrogen is needed for a similar increase in UTS. The maximum hydrogen content of the Ti metal core was less than 0.7±0.3 atom percent (H/Ti = 0.007±0.003). Hence, an increase in the strength of the core cannot account for our results.

In the second explanation, the tensile properties of the sheath are assumed to be of the same order of magnitude as those of α-Ti. Then the growth of the sheath at the expense of the α-Ti would cause little variation from the maximum values for the tensile properties. In this case, it is even possible for the hydride sheath properties to exceed those of α-Ti and show little or no increase beyond the maximum. Erosion could reduce the effective cross section of the sheath. Variations in the amount of erosion could account for part of the scatter in the data for specimens with H/Ti > 0.02.

In this explanation, we are assuming the hydride sheath contributes to the strength of the specimen. Therefore, the sheath must remain intact until fracture is imminent. It should also be noted that many of the specimens with H/Ti > 0.02 show elongations greater than the elongation of annealed uncharged specimens. Hence, we suggest that some of these hydride sheaths are somewhat ductile. This suggestion is in accord with a similar report of Kolachev and Livanov (ref. 2).

Possible Reasons for Property Differences in Titanium Hydride

If indeed some Ti hydride is ductile instead of being brittle as is generally believed (refs. 1 and 4), an explanation for the difference could lead to an important advance in this field. The difference may be due to either the impurity content or the hydrogen content of the hydride (i.e., γ-Ti) layer and/or its thickness.

We believe that the impurity content of our hydride is less than that of the brittle hydrides. The brittle hydrides were prepared by absorption of hydrogen gas at elevated temperatures. Our hydride was prepared at room temperature by electrolytic charging. We have already suggested that the high-temperature method would increase the oxygen and nitrogen content more than the electrolytic method, which was used at room temperature. The proof of this requires an effort in analytical chemistry that is beyond the scope of the present report; however, there is support for the contention that increases in oxygen and nitrogen content are almost unavoidable in hydriding of Ti at elevated temperature. Williams, Koehl, and Bartlett report that these impurities are preferentially adsorbed by specially cleaned Ti surfaces at room temperature even from hydrogen containing as little as 1 ppm oxygen and 5 ppm nitrogen (ref. 10). At elevated temperatures, these impurities would diffuse into the specimen to permit the adsorption of more oxygen and nitrogen. Korn and Zamir prepared very brittle Ti hydride by high-temperature absorption of hydrogen with 50 ppm impurities (ref. 4). Hence, we are convinced that extremely pure hydrogen that does not become contaminated
during the absorption process must be used to avoid an increase of these impurities during high-temperature absorption.

The difference in tensile properties of the hydrides might also be explained in terms of their hydrogen contents. Our hydride had an H/Ti near 1.2, while Korn and Zamir's hydrides with high hydrogen concentration (H/Ti > 1.81) were quite brittle (ref. 4). Hence, this explanation merits further investigation. If it is correct, then the hydrogen content of \( \gamma \)-Ti (Ti hydride), which can vary from H/Ti near 0.9 to 1.99 (ref. 12), must be known before an inference can be made concerning the ductility of a specific sample of \( \gamma \)-Ti.

Let us now consider possible effects of the thickness of the hydride sheath on its brittleness. A thin sheath may be ductile in a manner similar to the hydride lamellae of Kolachev and Livanov (ref. 2). As the \( \gamma \)-Ti sheath increases in thickness, the hydrogen content will tend to become greater near the outer surface than in the interior of the sheath. And if brittleness of Ti hydride is due to a high hydrogen content, then a relatively ductile hydride layer may lie between the brittle hydride and the metal core. The critical thickness and hydrogen content of the hydride layer are not known. Such behavior could also account for some of our scatter for the specimens with the thicker sheaths.

More work is required before any of these possible explanations for the difference in brittleness of Ti hydrides can be eliminated.

CONCLUDING REMARKS

Many of the tested specimens which had a completed hydride sheath \((0.02 < \frac{H}{Ti} \approx 0.33)\) were at least as ductile as annealed uncharged Ti. The YS and UTS of the same specimens were either equal to or greater than the values of the annealed uncharged Ti. These findings lead us to the conclusion that TiH\(_x\) \((x > 1)\) is not necessarily brittle or weak. The brittleness found in some hydrides may be caused by impurities such as oxygen and nitrogen or by a hydrogen content that is greater than that in the ductile hydrides.

A nonbrittle hydride sheath may form a protective coating against hydrogen diffusion into the core from gaseous hydrogen or from corrosion. This would be applicable where the sheath temperature was low enough for the sheath to be stable (probably \(T < 100\) C). A hydride sheath may also be protective for \(\alpha\)-stabilized Ti alloys; it is reported (ref. 7) that a hydride sheath does form on this type of Ti alloy.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 27, 1971,
114-03.
REFERENCES


TABLE I. - IMPURITY CONTENT OF TITANIUM WIRE PRIOR TO ANNEALING

<table>
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TABLE II. - SUMMARY OF CHARGING AND TENSILE DATA

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a At 8 mA/cm² except where noted.
b At 40 mA/cm².
Figure 1. - Micrographs of cross sections of specimens with varying values of atom ratio H/Ti. Sections were adjacent to analyzed portions.
Figure 2. - Tensile properties as function of bulk hydrogen concentration.
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