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Contents.

**INSTITUTE FOR THE STUDY OF FATIGUE,
FRACTURE AND STRUCTURAL RELIABILITY**

**MECHANICAL PROPERTIES AND MICROSTRUCTURE
OF HEAT TREATED AND QUENCHED Ti-6Al-4V.**

- A. The Fatigue Life of Ti-6Al-4V Tested at 260°C in the Annealed and as Quenched Condition;
- B. A Comparison of the Microstructure and Properties of Small Diameter Rod and Thick Plate;
- C. Corrosion-Fatigue Properties of the Ti-6Al-4V Alloy; and
- D. Microstructure and Properties of Ti-6Al-4V Heat Treated at 900°C and 927°C Followed by a Water Quench. *←*

By

C. M. Gilmore ~~and~~ M. A. Imam

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THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF
HEAT TREATED AND QUENCHED Ti-6Al-4V

PREFACE

In the previous year's report¹ we showed that in Ti-6Al-4V if the specimen was heat treated at about 900°C and water quenched, significant improvement in fatigue life was observed. We also presented evidence that showed that in the as quenched condition a significant amount of β phase had been retained and that upon subsequent cycling the β phase transformed to martensite. We proposed that the improved fatigue life was a result of strain energy being absorbed in transforming the β to martensite rather than producing fatigue damage.

During the present year we have tried to test the limitations of this mechanism for producing an improved fatigue life in Ti-6Al-4V. We wanted to see under what conditions we could expect to observe an improved fatigue life in Ti-6Al-4V when utilizing this mechanism to improve fatigue life. The experiments that will be reported are as follows:

- A. The fatigue life of Ti-6Al-4V tested at 260°C in the annealed and as quenched condition.
- B. A comparison of microstructure and properties of small diameter rod and thick plate.
- C. Corrosion-Fatigue properties of the Ti-6Al-4V alloy.

D. Microstructure and properties of Ti-6Al-4V heat treated at 900°C and 927°C followed by a water quench.

An interesting and important fundamental observation is evolving out of this rather diverse appearing set of experiments. There are indications that the martensite we are observing in the specimens heat treated at 900°C has isothermal character, meaning that the amount of martensite formed is a function of time. The martensite (α') of pure titanium has been shown to be athermal², meaning that the amount of martensite formed is dependent only upon the temperature of the quenching medium and not upon the cooling rate or upon time. It is also reported³ that the martensite (α') of titanium alloys is also athermal. We observe that this appears to be true for solution temperatures above the β transus. But as hold temperatures are decreased below the β transus the amount of martensite formed becomes time dependent upon the temperature and at 900°C (~ 80°C below the β transus) a significant fraction of β can be retained. The observation that the β phase can be retained at a heat treatment temperature of 900°C implies that this transformation could be isothermal; however, additional work will be necessary to confirm this. The other possibility is that the M_s and M_f temperatures have been suppressed to low temperatures. The experiments that indicate isothermal character will be discussed along with the experiments that were conducted to study the limitations of using the strain induced transformation of β to martensite to improve fatigue life.

The authors (CMG and MAI) express their appreciation to the National Bureau of Standards for use of their Transmission Electron Microscopy Facility, in particular the authors acknowledge the cooperation of Dr's Anna Fraker and William Ruff.

References

1. C. M. Gilmore and M. A. Imam: Microstructure of Solution Treated Ti-6Al-4V, Report III-B, The George Washington University, August 1976.
2. S. J. Wiskel, W. V. Youdelis and J. G. Parr: Trans. Met. Soc. A.I.M.E., 1959, 215, p. 875.
3. J. C. Williams: Proc. of the 2nd Int. Conf. on Titanium Science and Technology eds. R. I. Jaffee and H. M. Burte, Cambridge, Mass., May 1972, pp. 1433-1494.

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THE FATIGUE LIFE OF Ti-6Al-4V TESTED AT 260°C IN
THE ANNEALED AND AS QUENCHED CONDITIONS

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ABSTRACT

Fatigue tests were run on Ti-6Al-4V in the α - β annealed condition and after heat treatment at 900°C followed by a water quench. The fatigue tests were conducted by applying sinusoidal shear strain with maximum amplitude of 0.02 and $R=-1$ at 0.2 Hz. The tests were conducted at room temperature and 260°C. The α - β annealed material had twice the fatigue life at 260°C as it did at room temperature. This increase in life is due to the decrease in elastic modulus, this result would be expected in a fixed strain amplitude test. The heat treated material only had half the fatigue life at 260°C as it did at room temperature. This decrease in life is due to the rapid transformation of retained β to martensite that occurs at elevated temperature. The long life at room temperature has been attributed to the absorption of strain energy by the strain induced transformation of β to martensite. At elevated temperature it is proposed that the β to martensite transformation is thermally assisted and the β thus is more rapidly transformed to martensite resulting in a shortened fatigue life.

FATIGUE LIFE OF Ti-6Al-4V TESTED AT 260°C IN THE
ANNEALED AND AS QUENCHED CONDITIONS

INTRODUCTION

In the previous work we have shown that in the alloy Ti-6Al-4V, after a heat treatment at 900°C followed by a water quench, the room temperature fatigue life is increased relative to annealed specimens by more than an order of magnitude at a cyclic shear strain of 0.02.^{1,2} The fatigue life after heat treating at 900°C and quenching is at least four times that after solution treating at temperatures above the β transus. In titanium alloys if the temperature is above the β transus a single phase solid solution is formed, and the term solution treatment will be applied in this case; but below the β transus both α and β exist so the term heat treatment will be used for thermal treatments below the β transus.

The explanation of the observed increase in fatigue life is based upon transmission electron microscopy and diffraction data that indicates the specimens heat treated at 900°C have a significant amount of retained β after a water quench to room temperature. After cyclic loading the amount of martensite is significantly increased; it appears that a retained β to martensite transformation is induced by the cyclic strain and that the strain energy is absorbed in the

transformation and not in fatigue damage.

In this work we will study the effect of fatigue testing at elevated temperature upon specimens in the annealed and as quenched condition. The reason for this work is to see if the mechanism that produces an extended fatigue life at room temperature is also effective at elevated temperature.

EXPERIMENT

The material for this study was 1/4 inch diameter extruded rod of the composition shown in Table I. Specimens were cut to a length of 2.75 inches and then heat treated. One group of specimens was given an α - β anneal in a vacuum of at least 10^{-5} Torr.

α - β Anneal: 800°C for 3 hours, furnace cool to 600°C, air cool to room temperature.

The remaining specimens were heat treated in an air furnace for 10 minutes at 900°C and water quenched. After thermal treatment the specimens were machined and polished to a 0.05 μ m (alumina powder) surface with the dimensions shown in Figure 1. The specimens were torsion fatigue tested at room temperature and at 260°C in air with fully reversed ($R = -1$) sinusoidal strain cycle of maximum amplitude of 0.02 at a frequency of 0.2 Hz. The results of the fatigue tests are shown in Table II. Specimens for transmission electron microscopy were cut from the rods by spark discharge. The electropolishing was done in a solution

of 62.5% methanol, 31% butanol and 6.5% of perchloric acid (70% strength) at 13.9% volts and -40 to -50°C.

RESULTS AND DISCUSSION

The results in Table II show that the specimens heat treated at 900°C have a much different temperature dependence than the annealed specimens. The fatigue life of the specimens heat treated at 900°C decreases by approximately 50% when the temperature is increased to 260°C; whereas, the annealed specimens have almost twice the life when the temperature is increased to 260°C.

The result for the α - β annealed specimens can be understood when it is noted that these tests were conducted with a sinusoidal strain of maximum amplitude of 0.02. As the temperature increases the elastic modulus decreases (compliance increases), thus the specimen can more easily accommodate strain. It is generally known that with fixed strain cycling a more compliant structure will have a longer fatigue life. Therefore, with fixed strain amplitude cycling an increase in temperature should result in an increase in fatigue life as long as environmental degradation does not become more severe at the increased temperature, or as long as there is no change in the deformation mechanism at the increased temperature.

The above explanation of the increase in fatigue life at elevated temperature in the α - β annealed alloy obviously does not apply to the as quenched material because the fatigue life

decreases at elevated temperature rather than increases. Thus something must be happening in the quenched specimen that is not happening in the annealed specimen. It is not likely that there is any significant difference in environmental effects because there was no apparent difference in the condition of the specimens after testing. Rather we expect that the as quenched material is changing during the elevated temperature test. At room temperature we have proposed that the long fatigue life of the as quenched material is a result of the strain energy being absorbed in the strain induced martensitic transformation plasticity rather than in fatigue damage. This mechanism of plasticity requires the alloy to be in the retained β phase. We propose that holding the as quenched specimen containing retained β at an elevated temperature accelerates the strain induced transformation of retained β martensite. There is probably an interaction between the increased amount of thermal energy and the cyclic strain energy such that the thermal energy increases the rate of strain induced transformation of retained β to martensite. Because the specimen transforms to martensite more quickly at elevated temperature the mechanism that absorbs the plastic strain energy is consumed more quickly. Thus the as quenched specimens would be expected to have a shorter fatigue life at elevated temperature than at room temperature.

This conclusion is supported by transmission electron micrograph (TEM's) in Figures 2 and 3. Figure 2 is a TEM from the as quenched Ti-6Al-4V. The relatively dark bands are martensite and the light area between bands is retained β . Figure 3 is a TEM taken after cycling to failure at 260°C. It is obvious that the material that was retained β has extensively transformed to martensite. We propose that once the material is transformed to martensite then the fatigue life is just that of a martensitic microstructure which is shorter than that of a retained β microstructure.

CONCLUSIONS

1. The fatigue life of Ti-6Al-4V heat treated at 900°C and water quenched is greater than α - β annealed material at both room temperature and at 260°C.
2. For Ti-6Al-4V in the α - β annealed condition the fatigue life increases by a factor of 2 as the temperature is increased from room temperature to 260°C. The increased fatigue life at higher temperature is attributed to increased compliance in a fixed strain experiment.
3. For Ti-6Al-4V in the as quenched condition the fatigue life decreases as the temperature is increased. The decrease in fatigue life as temperature increases is attributed to a more rapid transformation of retained β to martensite.

REFERENCES

1. C. M. Gilmore and M. A. Imam: Report 3-B Microstructure of Solution Treated Ti-6Al-4V, The George Washington University, Washington, DC, August 1976.
2. M. A. Imam, C. M. Gilmore and M. D. Valentine: Improvements in Fatigue Life of Ti-6Al-4V, Second International Conference on Mechanical Behavior of Materials, Boston, August 1976, p. 552.

Table I. Chemical analysis of Ti-6Al-4V in weight percent

Al	V	O	C	N	Fe	H
6.4	4.0	.141	.01	.014	.18	55ppm

Table II. Fatigue test results for Ti-6Al-4V specimens tested at room temperature and at 260°C with a shear strain of ± 0.02 at 0.2 Hz.

Specimen Preparation	Test Temperature	Mean ¹ Life	Minimum Life	Standard Deviation
α - β Anneal	RT	944	429	443
α - β Anneal	260°C	1987	1557	894
H.T.-900°C+WQ	RT	9616	8917	758
H.T.-900°C+WQ	260°C	4818	4090	634

1. Experimental results are based upon four specimens for each test condition.

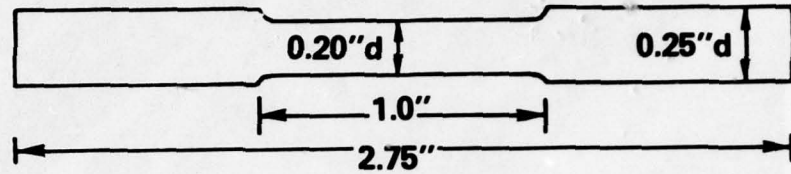


Figure 1. Fatigue specimen design.



Figure 2. Transmission electron micrograph of Ti-6Al-4V after heat treating at 900°C for 10 minutes followed by a water quench.



Figure 3. Transmission electron micrograph of the same material as in Figure 2 but after fatigue testing at 260°C.

A COMPARISON OF MICROSTRUCTURE AND
PROPERTIES OF SMALL DIAMETER ROD
AND THICK PLATE Ti-6Al-4V

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ABSTRACT

The microstructure, hardness and fatigue life of the alloy Ti-6Al-4V in the form of 1/4 inch diameter rod and 2 inch thick plate is compared. After heat treating at 900°C and water quenching, the thick plate material exhibited extensive surface relief; whereas, in the 1/4 inch diameter rod material little surface relief was observed. This indicates extensive martensite formation in the thick plate and less martensite formation in the 1/4 inch rod material. TEM observation confirmed extensive martensite formation in the thick plate. TEM observation of the 1/4 inch rod indicated parallel laths of martensite mixed with significant amount of retained β . The martensite formed in the thick plate material had a different morphology; the martensite laths intersected at angles of about 30°. The microstructure and hardness of the 2 inch thick plates was uniform across the section; and the hardness of the 2 inch thick plates was greater than that of the 1/4 inch diameter rod material.

This would be expected if the 2 inch thick plate had more martensite. The grain size of the 2 inch thick plate was 14.3 μm and that from the 1/4 inch diameter rods was 3.6 μm . The fatigue life of the specimens made from the 1/4 inch diameter rod was an order of magnitude greater than the specimens made from the 2 inch thick plate. This difference was attributed in part to the presence of retained β in the specimens made from 1/4 inch rod. During fatigue cycling the retained β has been observed to have a strain induced transformation to martensite. It is proposed that strain energy is absorbed in the deformation of transformation rather than fatigue damage. The material in the 2 inch thick specimens is already martensite and thus the transformation mode of deformation is not available. Other differences between these materials such as grain size chemistry and martensite morphology have yet to be investigated.

A COMPARISON OF MICROSTRUCTURE AND
PROPERTIES OF SMALL DIAMETER ROD
AND THICK PLATE Ti-6Al-4V

INTRODUCTION

In previous work we have shown that the fatigue life of Ti-6Al-4V can be significantly increased if the specimens are heat treated at about 900°C and water quenched.¹ We have chosen to use the terminology heat treatment rather than solution treatment because the latter implies that a single phase solid solution is created. In titanium alloys if the temperature is above the β transus, a single phase solid solution is formed and the term solution treatment will be applied in this case; but below the β transus both α and β exist so the term heat treatment will be used for thermal treatments below the β transus. The increase in fatigue life in the as quenched material has been attributed to the presence of a significant amount of retained β that transforms to martensite during cycling. The observation of retained β after quenching indicates that the β to martensite transformation under these conditions could be time dependent (isothermal) instead of time independent (athermal) as is generally observed for titanium alloys.² Our previous work was done on specimens with a small cross section of either 1/4 or 1/2 inch diameter. If the β to martensite transformation at this temperature is time dependent, slower cooling rates should result in more nucleation and growth of martensite. In thick sections of Ti-6Al-4V slower cooling rates

during quenching would be expected, thus we would expect more martensite if the transformation is time dependent; however, if the transformation of β to martensite is athermal, as is usually observed, then the slower cooling rate should have no effect on the amount of martensite formed. In this work we want to determine if the microstructure of a quenched thick plate is similar to a small rod. We also want to determine if the microstructure is uniform across the section of a thick specimen. In addition we will determine the fatigue properties of specimens made from the thick section material and compare these properties with specimens made from 1/4 inch diameter rod.¹

EXPERIMENTS AND RESULTS

The Ti-6Al-4V alloy for the thick section specimens was 2 inches thick with the composition shown in Table I; the plate was received in the mill annealed condition. The properties of the 2 inch thick plate material are compared with properties of extruded rod of 1/4 inch diameter whose composition is also given in Table I; the extruded rod was also received in the mill annealed condition. The 2 inch thick plate material was cut into a specimen 4"x4"x2". The plate was heat treated in an air furnace at 900°C. As shown in Figure 1 a hole was drilled to the specimen center and a thermocouple was inserted, the specimen was held in the furnace until the temperature reached 900°C, the temperature was then held constant for 20 minutes.

The thermocouple was then slowly withdrawn and the temperature profile was determined; there was a gradual increase of temperature to 907°C at the specimen surface. The specimen was then cut from its suspension in the furnace and allowed to freely drop into a water tank; so there was little delay between the furnace and the water quench.

The specimen was sectioned as shown in Figure 1 and hardness measurements were made in two perpendicular directions across the cut section. There was no significant variation in hardness across the section in either direction. The average hardness was $69.18 \pm .39$ Rockwell A. This hardness is a little higher than the average hardness of $66.76 \pm .69$ from the 1/4 inch diameter rod. These hardness results indicate that the properties of the thick block material should be uniform across the thick section, and the slightly higher hardness of the thick section indicates there may be some differences in the microstructure of the large and small specimens.

The microstructure of these specimens was characterized with optical, scanning electron (SEM) and transmission electron micrographs (TEM). SEM's were used to see if there was any significant variation in microstructure across the thick specimen. The SEM's presented here were all etched in 4% HF for 10 seconds. The material for the TEM's was spark discharge cut from the block. Thin foils were produced by electropolishing in a solution of 62.5% methanol, 31% butanol and 6.5% perchloric

acid (70% strength) at 13.9 volts and -40 to -50°C. Figure 2 is a SEM taken from the outside edge and its location is marked in Figure 1; Figure 3 was taken from the center of the specimen as located in Figure 1. Figures 2 and 3 show that there is little difference between the microstructure at the outside of the specimen and at the center of the specimen. Figures 2 and 3 show that this material is made up of two phases. The phase with light spots is the primary α (Hexagonal) phase. The darker phase is the phase that was β before the quench, and during the quench much of this phase appears to have transformed to martensite as is indicated by the presence of surface relief lines. A TEM from this specimen in Figure 4 confirms that much of the β phase transformed to martensite laths during the quench. Also in Figure 2 and 3 islands of α can be observed within the martensite grains.

The microstructure of the solution treated and quenched 1/4 inch rod is shown in the SEM of Figure 5 at a magnification similar to that in Figures 2 and 3. The grain size difference is immediately obvious, the grain size of the 1/4 inch rod is approximately 3.6 μm and the grain size of the thick plate is approximately 14.3 μm . It is necessary to view the 1/4 inch rod at higher magnification as in Figure 6 to make a comparison between the two materials. The magnification in Figure 6 was chosen to make the grains in this figure appear to be of similar size as those in Figures 2 and 3. Comparing the 1/4 inch rod

(Figure 6) with the 2 inch thick plate (Figures 2 and 3) shows that in spite of the grain size difference the microstructures look quite similar; each is a mixture of two phases, α grains (light) and grains that were previously β (dark). Also there are islands of α within the grains that were previously β in each material. However, close examination of the dark grains in each alloy shows that there is a difference in the dark grains. In the 2 inch thick specimen the dark phase (Figures 2 and 3) has many lines from surface relief indicating the presence of martensite. The dark phase in the 1/4 inch rod (Figure 6) has much less surface relief indicating much less martensite formation. This is more clearly shown in the higher magnification SEM's of Figures 7 and 8. The surface relief is clear in the 2 inch thick specimen (Figure 7) but in the 1/4 inch diameter rod most of the grains show little or no surface relief as is demonstrated in Figure 8. The TEM in Figure 9 from the 1/4 inch diameter rod confirms that the material that was β at the heat treatment temperature did not completely transform to martensite during quenching; a significant amount of β is retained mixed with fine martensite laths. Compare the TEM from the 1/4 inch diameter specimen (Figure 9) with that from the 2 inch thick specimen (Figure 4) and it is clear that the martensite has not formed in the 1/4 inch diameter specimen to the extent that it has in the 2 inch thick specimen. Also note that there is a significant difference in the morphology of

the two martensites, one is parallel laths the other intersecting laths.

Specimens for torsion fatigue testing were prepared in the shape shown in Figure 10. The specimens from the 2 inch thick plate were cut so that the axis of the specimen was transverse to the rolling direction. The specimens were tested at room temperature with a fully reversed ($R = -1$) sinusoidal shear strain of maximum amplitude of 0.02 at a frequency of 0.2 Hz. The results of the fatigue tests are presented in Table II. The fatigue life of the specimens taken from the 1/4 inch rod material is an order of magnitude greater than the specimens taken from the 2 inch thick plate.

DISCUSSION

There are two main points that will be discussed:

1. The difference in the amount of β that transformed to martensite in the 1/4 inch diameter and 2 inch thick specimens.
2. The difference in the fatigue life of the material from the 1/4 inch diameter rod and the material from the 2 inch thick plate.

There appears to be some controversy concerning the presence or absence of retained β following a treatment at 900°C with a water quench. For example, Averbach³ et. al. did not observe any retained β following heat treatments down to 800°C with water quenching. However, in their research the

specimens were initially solution treated at 1005°C and water quenched. The prior solution treatment temperature (1005°C) is above the β transus; thus the material should be totally α' after this quench. The subsequent heat treatments were at temperatures from 1005°C to 800°C. The β transus of Ti-6Al-4V is normally about 980°C, any subsequent heat treatment below the β transus should produce an equilibrium mixture of α and β . However, the subsequent heat treatment temperatures were only a small amount below the β transus. Thus the driving force for nucleation of β would be relatively small and the rate of nucleation and growth of the β phase could be very slow. Thus it is conceivable that no retained β phase was observed because at these heat treatment temperatures nucleation and growth of the β phase did not occur at a sufficient rate. Williams and Blackburn⁴ studied the phases present after heat treatment and quenching of Ti-6Al-4V and they found that retained β was present after most heat treatment temperatures except for temperatures from 860°C to 900°C. In their work the initial material was in the mill annealed, equiaxed condition. Thus the β phase was already present and growth of the β phase occurred at the heat treatment temperature but nucleation was not necessary. Fopiano⁵ et. al. performed some subsequent work where the initial material was rolled bar stock but the rolling temperature and the resultant microstructure was not given. This material was heat treated at temperatures from 1000°C to 750°C. However, no prior solution

treatment was given to these specimens. The results of these tests were essentially the same as Averbach's previous tests; no retained β was observed for heat treatment temperatures above 800°C. However, these results correlated with those of Williams and Blackburn are not clear. Griest⁶ et. al. also observed no retained β in Ti-6Al-4V of uncertain history following a heat treatment at 871°C and a water quench.

In our work we are beginning with mill annealed material as did Williams and Blackburn. The heat treatment at 900°C is about 80°C below the β transus; at equilibrium the alloy should be a mixture of α and β . Optical microscopy indicates that the α and β are in approximately equal volumes at 900°C. The amount of β at 900°C is increased relative to the amount following mill annealing at about 750°C. This increase in the amount of β phase would require a decrease in the amount of β stabilizer (V) because the vanadium must be spread through a greater volume. The decreased amount of β stabilizer will make the β phase less stable during quenching. If there is sufficient β stabilizer, the β phase can be retained upon quenching. This work shows that in our specimens a significant amount of the β phase could be retained in the 1/4 inch diameter rods. However, in the 2 inch thick specimens more of the β phase is transformed to martensite. The cooling rate during the quench is faster in the 1/4 inch rods and more of the β was retained rather than transforming to martensite. However, in the 2 inch thick

specimens with the slower cooling rate more of the β is transformed to martensite. This would imply that this particular transformation may be time dependent (isothermal). In other work related to this we have quenched specimens from higher solution temperatures such as 927°C and 955°C.⁷ The β phase when solution treated at these higher temperatures appears to fully transform to martensite even in the small diameter specimens. Thus it appears that the β can only be retained in significant amounts at lower solution temperatures such as 900°C; at higher temperatures β appears to transform to martensite. Either there is some temperature or temperature range where the β to martensite transformation changes from time independent to time dependent, or the β to martensite transformation in Ti-6Al-4V is in general time dependent and previous workers have not used sufficient cooling rates to retain β . It is quite likely that the transformation will be affected by the local composition of β stabilizer in the β phase at the time of the quench. It is likely that the disagreement in the literature concerning the presence or absence of retained β would be clarified if local chemical analysis was performed on the retained β or martensite phase. Our work on this aspect was presented previously and will be published separately.⁸ The only other alternative to the above conclusions is that the M_s to M_f temperature range is being depressed to low temperature; these alternatives will be investigated in the future.

Once the microstructure and the transformations are understood, we can begin to understand the results of the fatigue test. The fatigue life of the specimens made from the 1/4 inch diameter rod was an order of magnitude larger than the specimens machined from the 2 inch thick plate. One important difference between these two sets of specimens is that in the specimens made from 1/4 inch diameter rod there is a significant amount of retained β . In previous work we have observed that the retained β transforms to martensite during fatigue cycling and the deformation accommodated by the strain induced transformation to martensite appears to absorb strain energy that could otherwise produce fatigue damage. In the specimens machined from the two inch thick plate, most of the β phase transformed to martensite during the quench; so this strain absorption mechanism was not available during cycling of this material. There are also several other differences between these two sets of specimens that could also affect the fatigue life: grain size, martensite form and preferred orientation; additional research will be necessary to evaluate the effect of these additional variables upon the fatigue life and the phase transformation.

It is also possible to rationalize the results of the hardness test now that the microstructure has been discussed. The material in the 1/4 inch rod specimens has a slightly lower hardness. This would be expected if this material was deforming by a strain induced martensitic phase transformation during the

the hardness test. Thus we would expect the martensite in the thick specimens to be harder than the retained β in the 1/4 inch diameter specimens.

CONCLUSIONS

1. The microstructure of the alloy made from the 1/4 inch diameter rod was significantly different from that of the two inch plate. The material in the 2 inch thick plate appeared to have transformed to martensite based upon SEM and TEM observation, a quantitative analysis of the amount of retained β has yet to be performed. The martensite in the thick plate appeared as intersecting laths whereas in the 1/4 inch rod the martensite appeared as parallel laths. A significant amount of retained β could be observed in the 1/4 inch rod material.
2. The fatigue life of specimens made from as quenched 1/4 inch diameter rod was an order of magnitude greater than the fatigue life of specimens cut from the large quenched block.
3. The grain size of the material in the 1/4 inch rod was 3.6 μm whereas in the 2 inch thick plate the grain size was 14.3 μm .
4. The hardness and the microstructure of the 2 inch thick specimens was uniform across the section.

5. The hardness of the 2 inch thick specimen was greater than that of the 0.25 inch diameter rod, this would be expected if the 2 inch thick specimen had a greater amount of martensite than the 0.25 inch rod.

REFERENCES

1. C. M. Gilmore and M. A. Imam: Microstructure of Solution Treated Ti-6Al-4V, Report IIIB, The George Washington University, August 1976.
2. J. C. Williams: Proc. of the 2nd Int. Conf. on Titanium Science and Technology eds. R. I. Jaffee and H. M. Burte. Cambridge Mass., May 1972, pp. 1433-1494.
3. B. L. Averbach, M. F. Comerford and M. B. Bever: Trans. Met. Soc. of AIME 1959, Vol. 215, 682-685.
4. J. C. Williams and M. J. Blackburn: Trans. ASM 1967, Vol. 60, 373-383.
5. P. J. Fopiano, M. B. Bever and B. L. Averbach: Trans. ASM 1969, Vol. 62, 324-332.
6. A. J. Griest, J. R. Doig and P. D. Frost: Trans. Met. Soc. AIME, 1959, Vol. 215, 627-632.
7. C. M. Gilmore and M. A. Imam: To be published.
8. C. M. Gilmore and M. A. Imam: Microstructure and Prop. of Ti Alloys, AIME Atlanta, GA. April 1977.

Table I. Chemical composition of Ti-6Al-4V alloys
in weight %

a. 2 inch thick plate

Al	V	O	C	N	Fe	H
6.2	4.1	.168	.02	.017	.19	62 to 89 ppm

b. 1/4 inch diameter rod

Al	V	O	C	N	Fe	H
6.4	4.0	.141	.01	.014	.18	55 ppm

Table II. Fatigue test results for Ti-6Al-4V $\pm$ 0.02
shear strain

Specimen	Mean Life ¹	Minimum Life	Standard Deviation
1/4 in Diam.	9616	8917	758
2 in. plate	831	728	74.4

1. Based upon four specimens of each type.

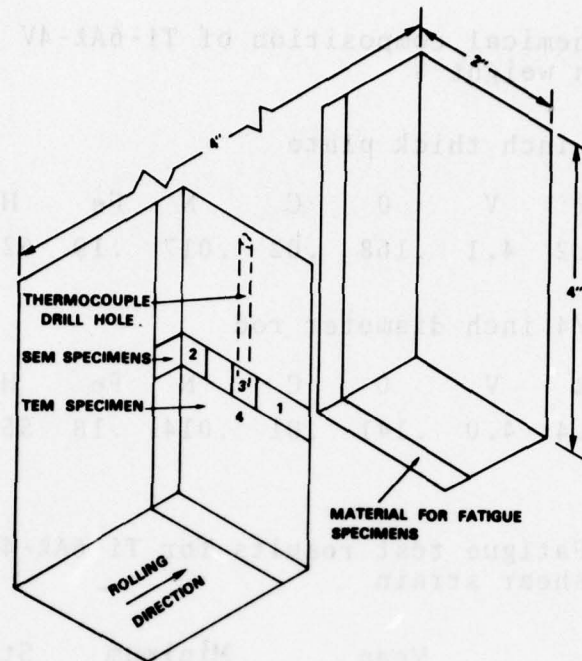


Figure 1. Configuration of the 4x4x2 inch specimen

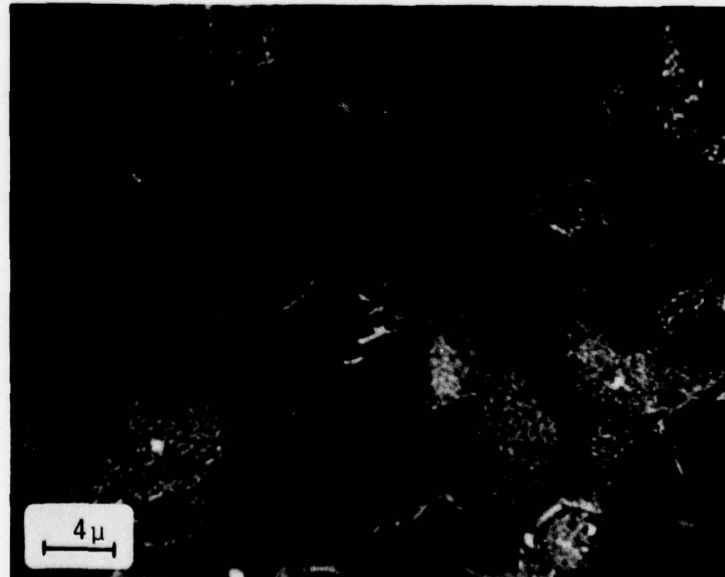


Figure 2. SEM taken at the outside edge of the thick specimen as noted in Figure 1.



Figure 3. SEM taken at the center of the thick specimen as noted in Figure 1. Note the surface relief.



Figure 4. TEM taken from the 2 inch thick specimen showing the martensite laths.



Figure 5. SEM of heat treated and quenched 1/4 inch diameter rod material.

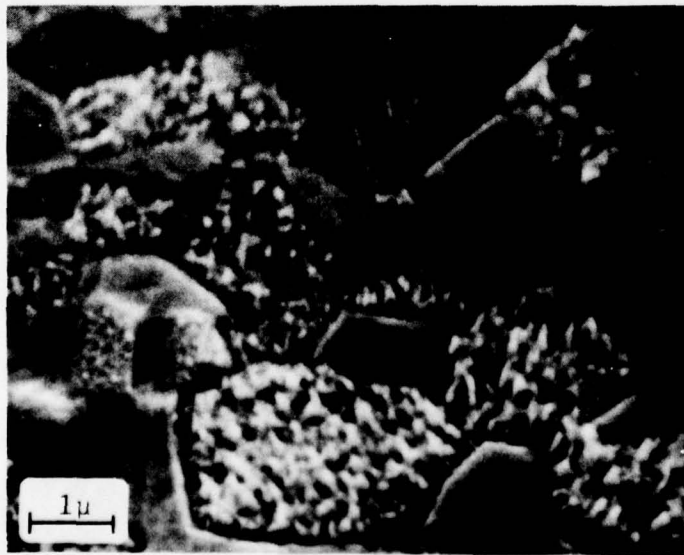


Figure 6. High magnification SEM of 1/4 inch diameter rod material.

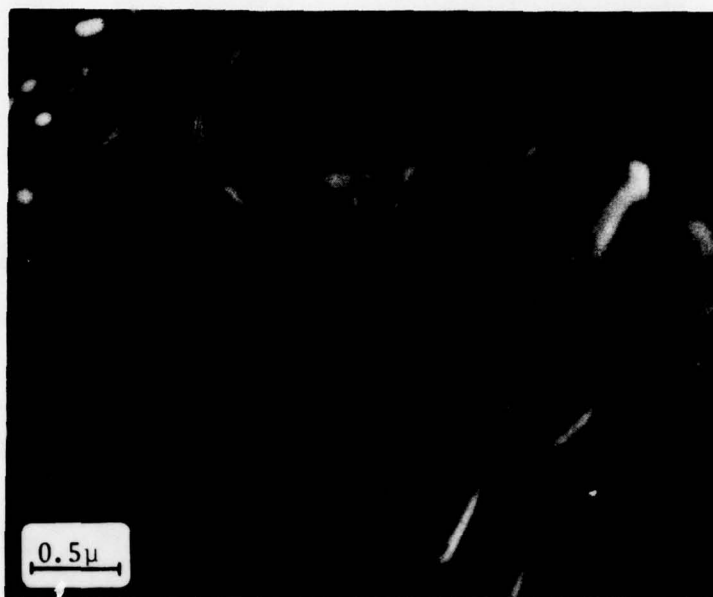


Figure 7. High magnification SEM of the thick specimen showing the large amount of surface relief.



Figure 8. Very high magnification SEM of the 1/4 inch diameter specimen showing the absence of surface relief. The white spotted grains are α , the uniform grey grains are retained β plus martensite.



Figure 9. TEM of 1/4 inch diameter rod showing the retained β with martensite laths. The martensite laths are the dark bands, the retained β is the light material between the bands, the large clear grains are α .

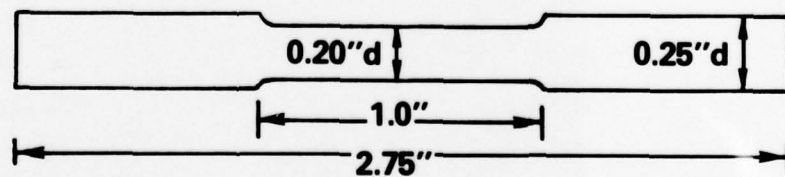


Figure 10. Fatigue test specimen design.

CORROSION-FATIGUE PROPERTIES OF THE Ti-6Al-4V ALLOY

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ABSTRACT

The high strain, low frequency corrosion-fatigue properties of the Ti-6Al-4V alloy in two different conditions were determined. One group of specimens was tested in the mill annealed condition (two hours at 704 and 760°C) and air cooled. Another group was heat treated at 900°C and water quenched prior to testing. Tests were conducted in Hanks' buffered saline solution at a temperature of 37°C and solution pH of 7.4. Specimens were subjected to fully reversed torsion fatigue in a flowing solution at a shear strain of 0.018 and a frequency of 1 Hz. The specimen electrode potential was monitored to follow the corrosion fatigue process. At the beginning of the corrosion-fatigue test, there is a drop in the electrode potential in the negative direction indicating the formation of many cracks in the oxide film. This

is followed by a rise in the positive direction and a leveling off of the potential at a much more negative value than the material would exhibit if there were no mechanical action indicating repassivation of some cracks while others continue to propagate. The results showed that the corrosion-fatigue life of the material heat treated at 900°C and water quenched is almost two times longer than that of the mill annealed material.

INTRODUCTION

Titanium and its alloys are highly corrosion resistant to saline solutions. Earlier work¹ investigating the corrosion-fatigue resistance of the Ti-6Al-4V alloy in saline solutions showed it to have superior corrosion-fatigue resistance when compared with 316 stainless steel or a Co-Cr-Mo alloy. This earlier work tested Ti-6Al-4V in the mill annealed condition. Recent studies² relating the Ti-6Al-4V alloy microstructure to fatigue life have shown that the mill annealed material when heat treated at 900°C (1650°F) and quenched can have as much as a ten fold increase in fatigue life when tested in air. The purpose of the present work was to determine whether this improvement in fatigue life remained when the material was fatigue tested in an aqueous saline environment and to study the general corrosion-fatigue behavior of the Ti-6Al-4V alloy. The tests were conducted using fully reversed torsion fatigue with the specimens immersed in a flowing saline solution. The results show that a corrosive environment adversely affects the fatigue life but that heat treating the mill annealed material at 900°C and quenching does improve the corrosion-fatigue behavior. Monitoring the electrode potential versus time during the fatigue tests indicated that cracks form and some are repassivated to a degree while others propagate to failure. This investigation shows that both the metal microstructure and the environment influence the corrosion-fatigue

behavior of the Ti-6Al-4V. Details of the work are as follows.

EXPERIMENTAL PROCEDURE

Specimens were made of ELI grade Ti-6Al-4V with the compositions shown in Table 1. The specimens were prepared from 0.65 cm. diameter rod cut to a 7.6 cm. length. A 1.27 cm. gauge section in the center was machined to a diameter 0.50 cm. Specimens were mechanically polished through a 0.05 μm alumina polishing powder to produce a smooth, scratch free surface. Following this, the specimens were ultrasonically cleaned in ethyl alcohol and then they were steam sterilized in an autoclave at 120°C and 15 p.s.i.

The environmental test cell for the corrosion-fatigue tests is shown in Figure 1. This cell was placed on the fatigue machine and tubes were connected to a pump which circulated the saline solution from a reservoir. The saline solution was Hanks' solution with a pH of 7.4, and a solution temperature of 37°C was maintained. The electrode potential of the specimen was measured versus a standard calomel electrode and was monitored with a strip chart recorder. Fatigue tests were conducted with an R value of -1 at a shear strain of 0.018 and a frequency of 1 Hz.

Microstructural analyses were conducted using methods of light microscopy, transmission and scanning electron microscopy and electron diffraction. Specimens for optical observation were mounted, mechanically polished through 0.05 μm alumina

polishing powder and etched with Kroll's etchant (3.5% HNO_3 , 1.5% HF and 95% H_2O). Thin foils for TEM were produced by electropolishing sections of the material which were cut from the bulk material by spark discharge cutting. The electropolishing was done in a solution 62.5% methanol, 31% butanol and 6.5% of 70% perchloric acid at 13.9 volts and -40 to -50°C.

Table 1. Chemical Composition of Ti-6Al-4V

Al	V	Fe	C	O	N	H	Ti
6.20	4.05	.15	.013	.10	.011	.0058	Remainder

RESULTS AND DISCUSSION

Microstructures

The microstructures of the annealed and heat treated condition of the Ti-6Al-4V alloy are shown in Figures 2 through 5. Three conditions of the alloy were studied. Two were in the mill annealed condition; one of these two had a two hour anneal at 704°C (1300°F) while the other had a two hour anneal at 760°C (1400°F). Light and transmission electron micrographs representative of the annealed materials are shown in Figures 2 and 3, respectively. These microstructures of the mill annealed materials consist mainly of α -Ti with a small amount of intergranular β -Ti. The third condition was produced by heat treating the mill annealed material for 10 minutes at 900°C (1650°F) and water quenching. Light and transmission electron micrographs of this heat treated material are shown in Figures 4 and 5, respectively. This microstructure can be described as

containing large α -Ti grains and martensitic areas of α' -Ti and β -Ti which surround areas of α -Ti. Selected area electron diffraction was used in the microstructural analysis.

Microscopic observation of the fractured specimen surfaces showed multiple crack initiation sites. A given fracture had both transverse and longitudinal breakage. Many cracks, parallel to the specimen axis, were present and they ran the entire length of the specimen.

Mechanical Tests

The corrosion-fatigue test results reported here were conducted at a high shear strain of ± 0.018 so that results could be obtained in a relatively short time. The corrosion-fatigue behavior will be sensitive to this aspect of the test as well as to the cycling frequency. The results of these tests, summarized in Table 2, show that under the conditions described in the experimental section, the material heat treated at 900°C and water quenched has a longer fatigue life than the material annealed at either 704°C or at 760°C . The fatigue life of the material which was annealed at 760°C is longer than that of the material which was annealed at 704°C . The average fatigue life of the annealed specimens is a little more than one-half of the average fatigue life of the heat treated specimens (900°C and quenched). The improvement in the heat treated and quenched material is even more striking if the minimum life of the annealed material, 1.713×10^4 cycles to failure, is compared

with the minimum life of the heat treated material which is 4.229×10^4 cycles to failure. Since there is some scatter in the corrosion fatigue data as indicated in Table 2, the values which should be used in the design and application of these materials are those of the minimum fatigue life.

Table 2. Corrosion Fatigue Life of Ti-6Al-4V in 10^4 Cycles for a Shear Strain of ± 0.018

Heat Treatment	Average Fatigue Life ¹	Minimum Fatigue Life	Standard Deviation
<u>Mill Annealed</u>			
a. 1300°F (704°C)	2.3283 ²	1.7130	-----
b. 1400°F (760°C)	3.5476 ²	3.2463	-----
c. TOTAL of a & b	2.938 ³	1.7130	±.7787
<u>Solution Treated</u>			
1650°F (900°C)	5.2504 ³	4.229	+1.0872

1. Number of cycles to failure
2. 2 specimens tested
3. 4 specimens tested

Electrochemical Measurements

Coupled with the mechanical action of fatigue in these tests was the corrosive environment. Some attempt was made to gain information relating to the formation of protective films on the metal in the solution and the cracking of the oxide film and the metal specimen. The measurement of the open circuit electrode potential of a metal specimen can give an indication of the formation or breakdown of the surface oxide film. If

the electrode potential vs. time rises in the positive direction, the indication is that a protective surface oxide film is forming. If the electrode potential rises and levels off, the surface oxide film has formed and remains intact. If the electrode potential is erratic, the surface film is unstable and probably pitting is occurring. Dropping of the electrode potential in the negative direction indicates dissolution of film, cracking of the film or other film breakdown which leads to corrosion. The open circuit potential vs. time curves for the two types of material used in this study are shown in Figures 6 and 7. In Figure 6, the dashed line shows the open circuit potential for the specimen in the corrosion-fatigue apparatus with no fatigue deformation. There is a rise in the positive direction and then the potential levels off. A film is formed on the metal which is stable in the saline solution at 37°C and this film remains intact. When the fatigue is started the electrode potential drops as shown in Figures 6 and 7. The magnitude of this drop in potential is dependent on the applied load of the fatigue test. This potential drop indicates cracks in the oxide film. The orientation of these cracks is related to the metal microstructure and the fatigue deformation. As the test progresses, some of these cracks become passivated with an oxide film again while other cracks remain exposed to the solution and as a result of the environment and the mechanical action are subject to propagation.

The repassivation of some of the cracks under the environmental and mechanical conditions of the test could account for the positive rise in the potential time curve and the leveling off of this curve. There is, again, a slight trend in the negative direction shortly before the specimen breaks. After failure, the electrode potential goes in the positive direction as (indicated by the arrows in these figures) and in the absence of fatigue, a surface film again covers the metal.

The general differences in electrode potential versus time curves for the mill annealed and quenched materials are shown in Figure 7. Shortly after the initiation of fatigue, there is an arrest in the electrode potential for the solution treated alloy and as time and fatigue progress, the potential drops, rises slightly and levels off at a value more negative than the mill annealed material. The delayed drop in the electrode potential for the 900°C material could be due to the absorption of mechanical damage by the microstructure which delays the breaking of the surface oxide film. Gilmore and Imam (3) have shown that in the solution treated microstructure the retained β phase transforms to α' during cycling. The applied strain is accommodated by a strain induced martensitic phase transformation rather than plastic deformation that produces fatigue damage. The strain accommodated during martensitic transformation appears to be less disruptive of the surface than the normal fatigue damage. In the solution treated alloy the surface oxide does eventually break and reformation of a

protective oxide does not appear to occur. In spite of this, the specimen life is significantly longer in the heat treated alloy under the conditions of this test.

CONCLUSIONS

It can be concluded from this study that the high resistance of Ti-6Al-4V to saline environmental corrosion and to mechanical fatigue is lowered when corrosion and mechanical action interact simultaneously. The influence of the metal microstructure is significant in corrosion-fatigue failure. The Ti-6Al-4V material in the mill annealed condition has a corrosion-fatigue life about one half as long as the same material in the heat treated (900°C) and quenched condition. The changes in open circuit potential in the mill anneal and heat treated specimens are different. In the heat treated specimen the potential drops more slowly and it remains at a low equilibrium value.

ACKNOWLEDGEMENTS

The ELI Ti-6Al-4V material was supplied by the Zimmer Co., USA, Warsaw, Indiana. The assistance of Donald L. Maske is acknowledged for specimen preparation and photographic work.

REFERENCES

1. Bowers, D. F., "Corrosion Fatigue: Type 304 Stainless Steel in Acid Chloride and Implant Metals in Biological Fluids," Ohio State University, Columbus, Ohio, 1975, Available from University Microfilms, Division of Xerox Corporation, Ann Arbor, MI.
2. Imam, M. A., Gilmore, C. M., and M. D. Valentine, "Improvement of Fatigue Life in Titanium Alloys," Second International Conf. on Mat., Boston, Mass., August 16-20, 1976.
3. Gilmore, C. M. and Imam, M. A., "Microstructure of Solution Treated Ti-6Al-4V," Institute for Fatigue, Fracture and Structural Reliability, Report III B, Contract No. N00019-76-C-0136, Available from the authors, The George Washington University.

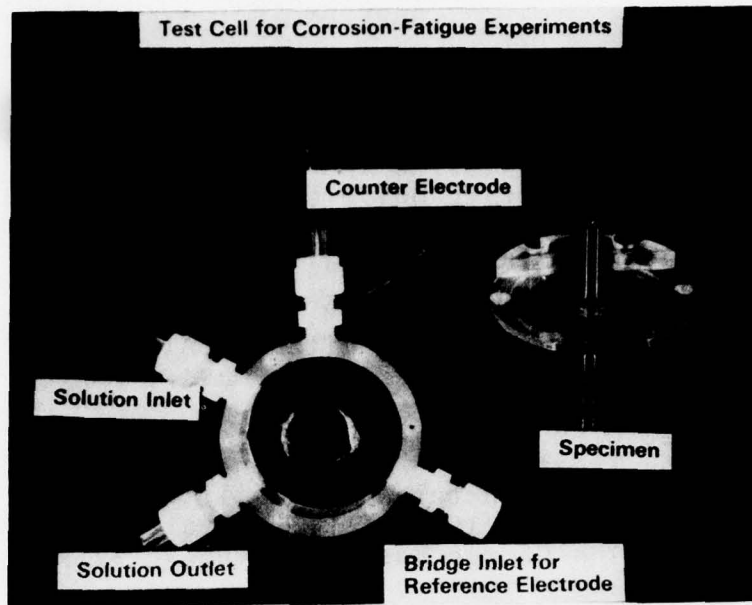


Figure 1. Test cell for corrosion-fatigue experiments.

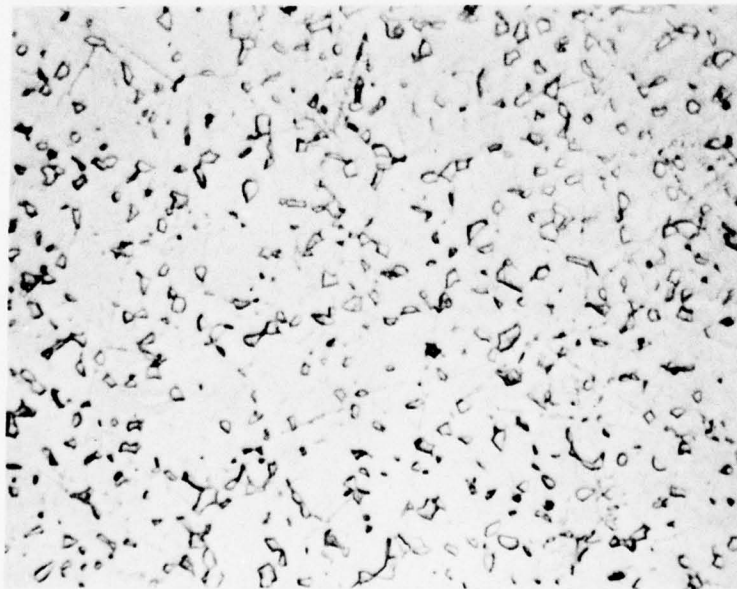


Figure 2. Light micrograph of Ti-6Al-4V mill annealed two hours at 760°C (1400°F). 1000X



Figure 3. Transmission electron micrograph of Ti-6Al-4V, mill annealed two hours at 760°C (1400°F)

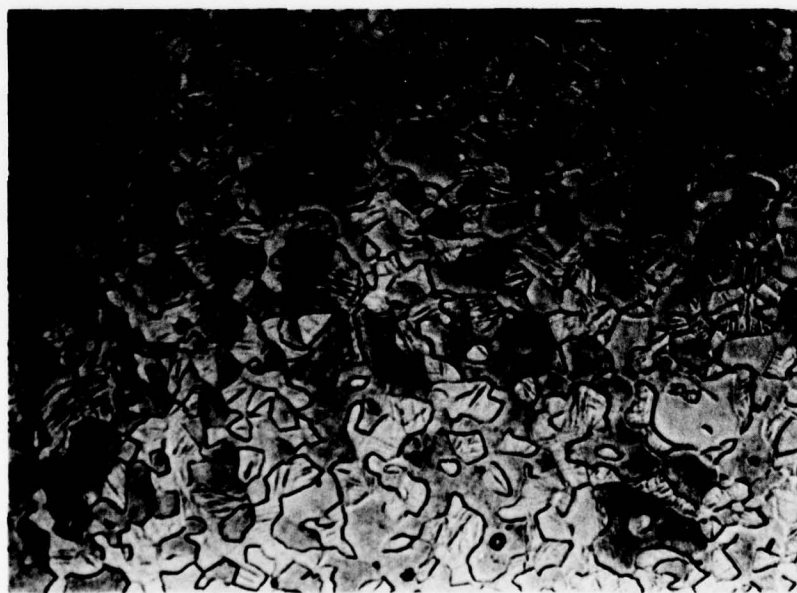
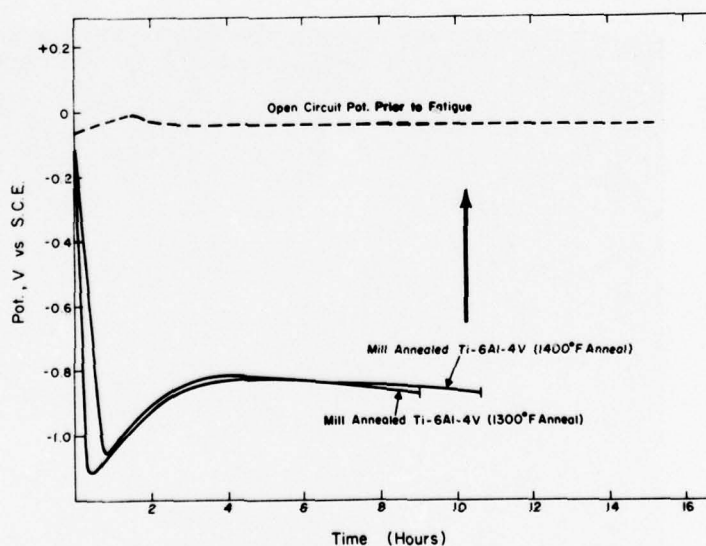


Figure 4. Light micrograph of Ti-6Al-4V heat treated 10 minutes at 900°C (1650°F). 1000X

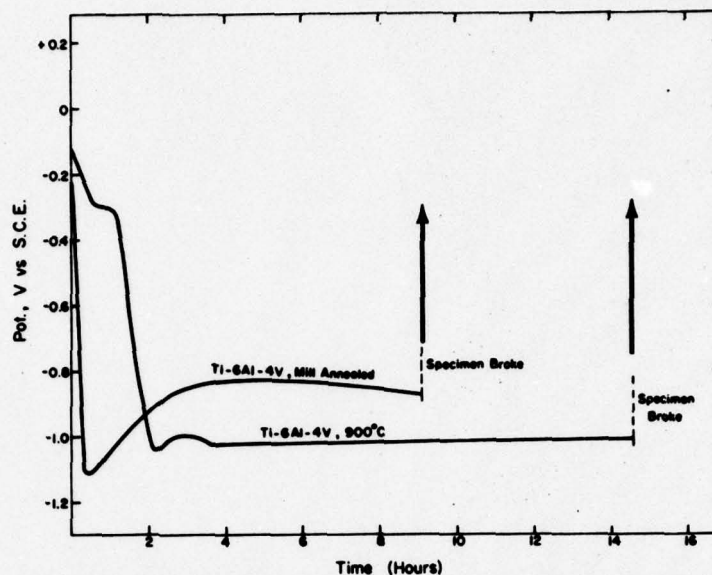


Figure 5. Transmission electron micrograph of Ti-6Al-4V heat treated 10 minutes at 900°C (1650°F).



Open circuit potential vs. time curves for Ti-6Al-4V, as indicated, during corrosion-fatigue testing (reversible torsion) in Hanks' solution (pH = 7.4) at 37°C.

Figure 6. Open circuit potential vs. time curves for Ti-6Al-4V, as indicated, during corrosion-fatigue testing (reversible torsion) in Hanks' solution (pH = 7.4) at 37°C.



Open circuit potential vs. time curves for Ti-6Al-4V as indicated during corrosion-fatigue testing (reversible torsion) in Hanks' solution (pH = 7.4) at 37°C.

Figure 7. Open circuit potential vs. time curves for Ti-6Al-4V as indicated during corrosion-fatigue testing (reversible torsion) in Hanks' solution (pH = 7.4) at 37°C.

MICROSTRUCTURE AND PROPERTIES OF Ti-6Al-4V
HEAT TREATED AT 900°C AND 927°C
FOLLOWED BY A WATER QUENCH

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ABSTRACT

Ti-6Al-4V alloy was heat treated at 927°C followed by water quench. The aim of this particular heat treatment was to see changes, if any, in microstructure and mechanical properties as compared to the heat treatment at 900°C followed by water quench.

Tensile test results of specimens heat treated at 927°C indicate higher hardness, yield stress and ultimate strength, but less ductility as compared to specimens heat treated at 900°C. Fatigue test results show a decrease in fatigue life as compared to the specimens heat treated at 900°C. SEM and TEM studies reveal the presence of two phases: an α -phase and a phase with martensite. The martensitic area also shows the presence of retained β but the percentage of retained β relative to the amount of α is less as compared to the heat treatment at 900°C. The lesser amount of retained β for a 927°C heat treatment is responsible for its lower fatigue life as compared to the heat treatment at 900°C.

MICROSTRUCTURE AND PROPERTIES OF Ti-6Al-4V
HEAT TREATED AT 900°C AND 927°C
FOLLOWED BY A WATER QUENCH

INTRODUCTION

In the previous work¹ we have shown the effect of heat treatments at 843°C, 900°C, 954°C and 1065°C followed by a water quench on fatigue lives. Among these treatments those specimens heat treated at 900°C and water quenched exhibited the longest life. Microstructural investigation of the alloy heat treated at 954°C or 1065°C and water quenched showed martensite only. Quenching after heat treatment at 900°C revealed a microstructure consisting of α -Ti grains and grains with martensite. The martensite grains contain islands of α -Ti plus retained β . In this work we wish to find out the changes, if any, in microstructure and mechanical properties if we increase the heat treatment temperature from 900°C to 927°C followed by water quench. We have chosen to use the terminology heat treatment rather than solution treatment because the latter implies that a single phase solid solution is created; whereas, heating to temperatures below the β transus ($\sim 990^\circ\text{C}$) results in chemical separation into two phases.

EXPERIMENTAL

Fatigue specimen blanks were cut to 2.75 inch length from 0.25 inch diameter extruded and mill annealed rod whose composition

is given in Table I. The specimen blanks were heat treated in an air furnace for 10 minutes at a temperature of 927°C and 900°C then water quenched. The specimens were machined to the approximate specimen dimensions shown in Figure 1. After machining, the gage section of the specimens was polished to 0.05 μm (alumina powder) surface. The specimens were torsion fatigue tested at room temperature using fully reversed ($R = -1$) sinusoidal shear of maximum amplitude 0.02 and frequency of 0.2 Hz. The results of the fatigue tests are presented in Table III.

Specimens for tensile tests were prepared from 5/4 inch diameter mill annealed rod according to the approximate specimen dimensions shown in Figure 2. The composition of 5/4 inch diameter mill annealed rod is given in Table II. The results of tensile tests are reported in Table IV. Specimens for microhardness measurements and scanning electron microscopy studies were prepared separately by electropolishing a suitable section of 0.25 inch rod in a solution of 55% methanol, 38.6% butanol and 8.4% perchloric acid at 14 volts and -20 to -25°C temperature. Microhardness measurements were made with a Wilson Tukon microhardness tester using a Knoop indenter with a load of 100 gms. The result of microhardness measurements are reported in Table V.

For transmission electron microscopy studies, a thin section was cut by spark cutter and electropolished in a solution of 62.5% methanol, 31% butanol and 6.5% perchloric

acid (70% strength) at 13.9 volts and -40 to -50°C temperature.

RESULTS AND DISCUSSION

The test results indicate higher hardness, yield stress and higher ultimate strength but less ductility after heat treatment at 927°C as compared to specimens heat treated at 900°C. Fatigue test results show a decrease in fatigue life for heat treatment at 927°C as compared to the heat treatment at 900°C.

Scanning electron microscopy studies of the material heat treated at 927°C indicate the presence of α -phase and martensitic phase as shown in Figure 3. This result is confirmed by transmission electron microscopy studies as shown in Figure 4. Further examination of the martensitic area under transmission electron microscope indicates the presence of retained β . Figure 5 is the TEM micrograph of the specimen heat treated at 900°C. Comparative study of Figures 4 and 5 for retained β shows that the amount of retained β in specimen heat treated at 927°C is less. Moreover the martensitic laths are thicker as compared to the specimens heat treated at 900°C.

It has been shown¹ that in the specimens heat treated at 900°C and water quenched, the retained β phase transforms to martensite during cycling. The applied strain is accommodated by a strain induced martensitic phase transformation rather than plastic deformation that produces fatigue damage. Since the amount of retained β in the specimens heat treated at

927°C is less, it experiences less strain induced martensitic transformation as compared to the specimen heat treated at 900°C. This could explain why the fatigue life of the material heat treated at 900°C (contains more retained β) is longer than the material heat treated at 927°C followed by water quench.

CONCLUSIONS

1. The hardness, yield stress and ultimate strength of specimens heat treated at 927°C compared to the specimens heat treated at 900°C is higher whereas the ductility is lower.
2. The fatigue life of the specimens heat treated at 927°C is lower than the fatigue life of the specimens heat treated at 900°C.
3. The specimen heat treated at 927°C and water quenched reveals under TEM the presence of an α -phase and a martensite phase. Further examination in TEM of the martensitic area shows the presence of retained β . The total amount of retained β is found to be less than that found in specimens heat treated at 900°C.
4. Lesser amount of retained β appears responsible for lower fatigue life of the specimen heat treated at 927°C as compared to the specimens heat treated at 900°C.

REFERENCE

1. C. M. Gilmore and M. A. Imam: Microstructure of solution treated Ti-6Al-4V, Report III B, The George Washington University, August 1976.

TABLE I

CHEMICAL COMPOSITION OF Ti-6Al-4V ALLOY USED FOR
FATIGUE TESTS AND MICROHARDNESS MEASUREMENT
IN WEIGHT PERCENT

Al	V	O	C	N	Fe	H
6.4	4.0	0.141	0.01	0.014	0.18	55 ppm

TABLE II

CHEMICAL COMPOSITION OF Ti-6Al-4V ALLOY USED FOR
TENSILE TESTS IN WEIGHT PERCENT

Al	V	O	C	N	Fe	H
6.3	4.3	0.188	0.02	0.010	0.17	67 ppm

TABLE III

FATIGUE LIFE OF Ti-6Al-4V SPECIMEN
CYCLED AT ± 0.02 SHEAR STRAIN

Thermal Treatment	Mean*	Minimum	Standard Deviation
S. T. -- 10 min. 927 C + W.Q	2223	2142	60.5
S. T. -- 10 min. 900 C + W.Q	9616	8917	758

* Results based upon four specimens of each thermal treatment

TABLE IV

TENSILE TEST RESULTS FOR Ti-6Al-4V

Thermal Treatment	Yield Strength 10^8 N/m^2 (Ksi)	Ultimate Strength 10^8 N/m^2 (Ksi)	Young's Modulus 10^{11} N/m^2 (10^7 psi)	Percentage Reduction in area	Percentage Elongation
S. T. -- 10 min. 927 C + W.Q*	10.12 (146.9)	11.84 (171.7)	1.061 (15.4)	32.54*	21.5
S. T. -- 10 min. 900 C + W.Q	9.99 (144.9)	11.59 (168.1)	1.20 (1.74)	39.8	26.0

* Average of two tests

+ Deviation in this Data was large

 $RA_1 = 28.33\%$ $RA_2 = 36.76\%$

TABLE V

KNOOP MICROHARDNESS RESULTS (LOAD = 100 GMS)

Thermal Treatment	Mean*	Minimum	Standard Deviation
S. T. -- 10 min. 927 C + W.Q	477	465	6.6
S. T. -- 10 min. 900 C + W.Q	455	450	7.1

* Results for Eight Measurements

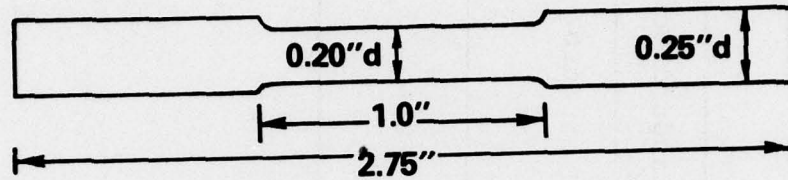
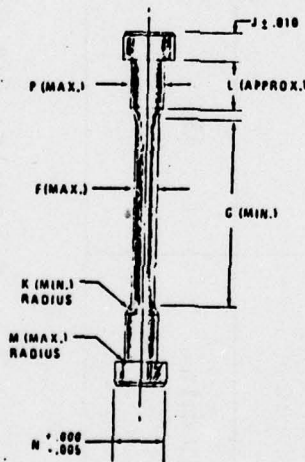


Figure 1. Fatigue specimen design.



Specimen Dimensions — Inches							
F	G	J	K	L	M	N	P
0.50	2.25	0.50	0.38	0.50	0.05	0.875	0.625

Figure 2. Tensile test specimen design.

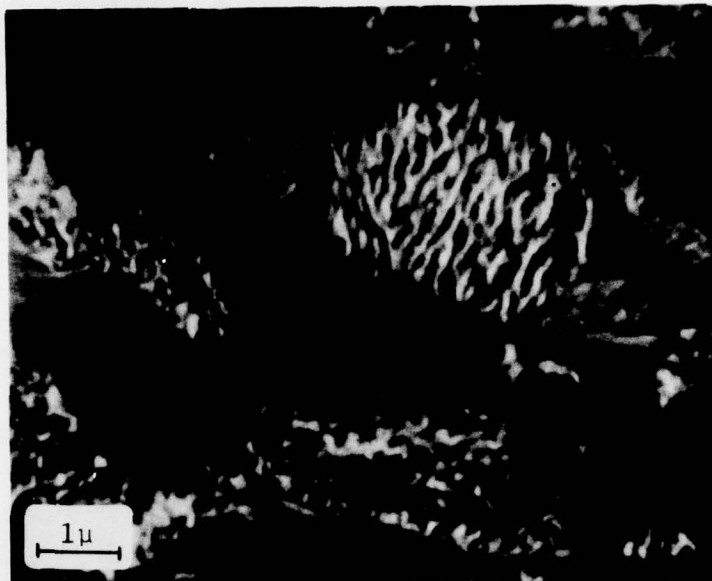


Figure 3. Scanning electron micrograph of Ti-6Al-4V, heat treated at 927°C for 10 minutes and water quenched. The light areas are primary α whereas the dark areas contain needles of α' (martensite) and retained β .



Figure 4. Transmission electron micrograph of Ti-6Al-4V, heat treated at 927°C for 10 minutes and water quenched. The light areas are primary α whereas the dark areas contain needles of α' (martensite) and retained β .



Figure 5. Transmission electron micrograph of Ti-6Al-4V, heat treated at 900°C for 10 minutes and water quenched. The light areas are primary α whereas the dark areas contain needles of α' (martensite) and retained β .

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This increase in life is due to the decrease in elastic modulus, this result would be expected in a fixed strain amplitude test. The heat treated material only had half the fatigue life at 260°C as it did at room temperature. This decrease in life is due to the rapid transformation of retained β to martensite that occurs at elevated temperature. The long life at room temperature has been attributed to the absorption of strain energy by the strain induced transformation of β to martensite. At elevated temperature it is proposed that the β to martensite transformation is thermally assisted and the β thus is more rapidly transformed to martensite resulting in a shortened fatigue life.

B. A Comparison of Microstructure and Properties of Small Diameter Rod and Thick Plate Ti-6Al-4V

The microstructure, hardness and fatigue life of the alloy Ti-6Al-4V in the form of 1/4 inch diameter rod and 2 inch thick plate is compared. After heat treating at 900°C and water quenching, the thick plate material exhibited extensive surface relief; whereas, in the 1/4 inch diameter rod material little surface relief was observed. This indicates extensive martensite formation in the thick plate and less martensite formation in the 1/4 inch rod material. TEM observation confirmed extensive martensite formation in the thick plate. TEM observation of the 1/4 inch rod indicated parallel laths of martensite mixed with significant amount of retained β . The martensite formed in the thick plate material had a different morphology; the martensite laths intersected at angles of about 30°. The microstructure and hardness of the 2 inch thick plates was uniform across the section; and the hardness of the 2 inch thick plates was greater than that of the 1/4 inch diameter rod material. This would be expected if the 2 inch thick plate had more martensite. The grain size of the 2 inch thick plate was 14.3 μm and that from the 1/4 inch diameter rods was 3.6 μm . The fatigue life of the specimens made from the 1/4 inch diameter rod was an order of magnitude greater than the specimens made from the 2 inch thick plate. This difference was attributed in part to the presence of retained β in the specimens made from 1/4 inch rod. During fatigue cycling the retained β has been observed to have a strain induced transformation to martensite. It is proposed that strain energy is absorbed in the deformation of transformation rather than fatigue damage. The material in the 2 inch thick specimens is already martensite and thus the transformation mode of deformation is not available. Other differences between these materials such as grain size chemistry and martensite morphology have yet to be investigated.

C. Corrosion-Fatigue Properties of the Ti-6Al-4V Alloy

The high strain, low frequency corrosion-fatigue properties of the Ti-6Al-4V alloy in two different conditions were determined. One group of specimens was tested in the mill annealed condition (two hours at 704 and 760°C) and air cooled. Another group was heat treated at 900°C and water quenched prior to testing. Tests were conducted in Hank's buffered saline solution at a temperature of 37°C and solution pH of 7.4. Specimens were subjected to fully reversed torsion fatigue in a flowing solution at a shear strain of 0.018 and a frequency of 1 Hz. The specimen electrode potential was monitored to follow the corrosion fatigue process. At the

Unclassified continue

beginning of the corrosion-fatigue test, there is a drop in the electrode potential in the negative direction indicating the formation of many cracks in the oxide film. This is followed by a rise in the positive direction and a leveling off of the potential at a much more negative value than the material would exhibit if there were no mechanical action indicating repassivation of some cracks while others continue to propagate. The results showed that the corrosion-fatigue life of the material heat treated at 900°C and water quenched is almost two times longer than that of the mill annealed material.

D. Microstructure and Properties of Ti-6Al-4V Heat Treated at 900°C and 927°C Followed by a Water Quench

Ti-6Al-4V alloy was heat treated at 927°C followed by water quench. The aim of this particular heat treatment was to see changes, if any, in microstructure and mechanical properties as compared to the heat treatment at 900°C followed by water quench.

Tensile test results of specimens heat treated at 927°C indicate higher hardness, yield stress and ultimate strength, but less ductility as compared to specimen heat treated at 900°C. SEM and TEM studies reveal the presence of two phases: an α -phase and a phase with martensite. The martensitic area also shows the presence of retained β but the percentage of retained β relative to the amount of α is less as compared to the heat treatment at 900°C. The lesser amount of retained β for a 927°C heat treatment is responsible for its lower fatigue life as compared to the heat treatment at 900°C.

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