

INFLUENCE OF TEMPERATURE ON HIGH CYCLE FATIGUE PROPERTIES OF TI-6AI-2Sn-4Zr-6Mo

Hodi

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#### ABSTRACT

High cycle fatigue data of unnotched Ti-6Al-2Sn-4Zr-6Mo revealed no change in fatigue strength at 900 F compared to room temperature, 400 F, and 700 F. At 1000 F, the decrease in fatigue strength from that at 900 F and below becomes noticeable. At 1200 F the decrease is significant. The fatigue strength-life curve of specimens coated with electroless nickel and diffusion bonded, which were tested at 1000 F, falls midway between results of uncoated specimens tested at 1000 F and 1200 F. Microstructural changes occurring with time, at temperature, indicate a refinement of structure. Fractography reveals three distinct areas: an initial radial area which is a mixed mode of quasi-cleavage and intergranular fracture; followed by a radial fibrous zone, the result of dimpled fracture; and finally a shear lip which occurs by shear fracture.



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#### INTRODUCTION

Continuing development of Army aircraft gas turbine engines is paced by a combination of material improvements and mechanical design. These factors are of particular importance for both axial and centrifugal rotors where the requirement is for good high cycle fatigue properties because of the relatively long, slender airfoils. However, further engine developments will mean higher shaft speeds and higher pressure ratios, hence, higher peak compressor temperatures. For these reasons, the materials currently in use (iron-base superalloys and Ti-6Al-4V) have a likely probability of being replaced due to their relatively low peak operating temperature capability. Army engines now in some state of development have peak compressor temperatures in the range of 700 F to 900 F.

Fatigue-type failures are a constant concern in critical material applications such as aircraft gas turbines. One source of fatigue is vibration, a consequence of rotation and/or fluid motion. Failure may result after a large number of stress cycles. Another prime source of fatigue is a consequence of start and stop operations, where the failure mechanism has been identified as thermal or low cycle fatigue. Here strain is the controlling variable, induced mostly by temperature effects such as thermal mismatch, temperature transients, etc.

The high fatigue strength-to-weight ratio, notch toughness, and corrosion resistance of titanium alloys have attracted considerable attention and interest in the hope of using titanium in dynamic aircraft compressor components. Since Army aircraft turbine engines must survive in an erosion/corrosion environment, it is likely that any titanium alloy used will require a coating. The effect of an electroless nickel coating has been determined to deteriorate the room temperature fatigue resistance of titanium alloys.<sup>1</sup> Electroless nickel is an underlayer for an erosion-resistant coating which is quite successful. Since the underlayer by itself degrades room temperature fatigue strength, its effect on fatigue resistance of titanium alloys at expected operating temperatures should be documented. However, data are unavailable on the effect of this coating on the fatigue resistance of titanium alloys at expected operating temperatures.

New or improved alloys for use in aircraft gas turbines are constantly being sought. There is a current interest in using the alpha/beta alloy Ti-6Al-2Sn-4Zr-6Mo for the impeller, taking advantage of its high yield strength and creep resistance at temperatures around 800 F. The fatigue properties for this alloy are available up to a temperature of 700  $F^2$ . The objectives of this investigation are to determine if the high fatigue resistance reported for this alloy at 700 F and below exists at the higher temperatures of interest to engine designers (900 to 1200 F), and determine the effect of coatings, such as electroless nickel, on the fatigue properties of Ti-6Al-2Sn-4Zr-6Mo alloy.

2. Titanium Alloys Handbook, MCIC-HB-02, December 1972, p. 5-7: 72-10.

<sup>1.</sup> LEVY, M., and MORROSSI, J. L. Erosion and Fatigue Behavior of Coated Titanium Alloys for Gas Turbine Engine Compressor Applications. Army Materials and Mechanics Research Center, AMMRC TR 76-4, February 1976.

#### MATERIALS AND EXPERIMENTAL PROCEDURE

The material tested, Ti-6Al-2Sn-4Zr-6Mo bar, was beta rolled and mill annealed (1300 to 1350 F for 2 hours and air cooled). The ingot chemistry in weight percent is:

<u>A1</u>	Sn	Zr	Mo	0	N	<u> </u>	Fe	H	Ti	
5.8	2.0	4.1	5.6	0.006	0.009	0.027	0.09	0.001	Bal.	

Specimens were machined from one-inch round bars into modified 426A tensile fatigue specimens (Figure 1). The modified specimen has oversized threads and a decreased cross-sectional area in the gage section. These modifications were necessary since during previous testing the standard specimen broke frequently in the threads, a common occurrence with high strength materials. The majority of tests were accomplished on a Sonntag Universal fatigue testing machine, Model SF10-U. Heating of the specimens was accomplished by a Satec three-zone split furnace, Model SF-15, controlled to  $\pm$  5 F. Fatigue tests were conducted at 900 F, 1000 F, and 1200 F, at a frequency of 30 Hz and a minimum to maximum stress ratio (R) of 0.1. Fractured specimens were sectioned parallel to the tensile axis and the microstructures examined near the fracture surface. Microstructures were also compared along the stress versus life curve for each temperature. Several fatigue tests were repeated at 900 F at a 30 Hz frequency on an Instron dynamic cycler, Model 1211. The Instron tester is hydraulically operated and, unlike the Sonntag tester, cycling can be stopped almost immediately without fracture surfaces coming into contact and destroying themselves. Replicas of the undamaged fracture surfaces were prepared for examination under a transmission electron microscope.



Modified Type 426A



One set of specimens with a diffusion-bonded electroless nickel coating was tested at 1000 F to determine the effect of the coating on fatigue life. Preparation of the test specimen involved the following sequence of operations: vapor degreasing, vapor blasting, activation, plating, aging, and heat treating.

Samples were degreased with trichlorethylene vapor and vapor blasted with 100-grit glass beads at 70 to 80 psig. The washed and cleaned specimens were immediately immersed in an activating solution of hot (150 F), slightly acidic, 10% nickel chloride solution for 2 minutes and transferred to a modified Brenner-Riddell plating bath.<sup>3,4</sup>

The plating bath composition is as follows:

Chemical	Formula	Grams	Moles	
Nickel Chloride	NiCl <sub>2</sub> •6H <sub>2</sub> O	30	0.126	
Ammonium Chloride	NH4C1	50	0.934	
Sodium Chloride	NaC1	5	0.085	
Sodium Citrate	Na 3C6H503 • 2H20	100	0.340	
Sodium Hypophosphite	NaH2PO2 · H2O	10	0.094	

All chemicals used were of reagent grade.

The plating conditions were: pH range 8 to 9; temperature 190 F  $\pm$  2 F; agitation constant with argon gas; plating time 2 hours, with an expected deposit of 0.5 to 0.6 mil of electroless nickel. Plated specimens were rinsed in running water, dried, and stored in a desiccator for at least 24 hours prior to heat treatment. The coating was subsequently vacuum diffusion bonded at  $10^{-4}$  torr and a temperature of 950 F for 4 hours, then furnace cooled in vacuum.

#### RESULTS

The experimental results are summarized in Table 1. Figure 2 shows stress versus lifetime cycles obtained at 900 F, 1000 F, and 1200 F compared with similar curves (taken from the Titanium Alloys Handbook<sup>2</sup>) of tests conducted at room temperature (RT), 400 F, and 700 F. Within the experimental spread, data at 900 F revealed no change in fatigue strength. At 1000 F, the decrease in strength capability from that at 900 F is 7 ksi at  $10^5$  cycles and 10 ksi at  $10^6$  cycles. However, at 1200 F there is a significant decrease in fatigue strength. The reduction is now 62 ksi at  $10^5$  cycles and 80 ksi at  $10^6$  cycles over the same lifetime cycle range.

Specimens coated with diffusion-bonded electroless nickel and tested at 1000 F had a fatigue life curve midway between that for uncoated specimens tested at 1000 F and 1200 F (Figure 2).

U.S. Patent No. 2,253,283, December 5, 1950.
 Symposium on Electroless Nickel Plating. American Society for Testing and Materials, Special Technical Publication No. 265, 1959.

			Str			
Specimen	Test Temp. (deg F)	Maximum	Mean	$\frac{\text{Alternating}}{\left(\frac{\max \ \alpha - \min \ \alpha}{2}\right)}$	Cycles	Break*
Uncoated	900	95 95 100 105 105 110 110	52.2 55.0 57.7 57.7 60.5 60.5	42.8 42.8 45.0 47.3 47.3 49.5 49.5	1.0 x 10 <sup>7</sup> 1.2 x 10 <sup>7</sup> 3.7 x 10 <sup>5</sup> 5.86 x 10 <sup>6</sup> 1.43 x 10 <sup>6</sup> 1.7 x 10 <sup>4</sup> 3.02 x 10 <sup>5</sup>	Runout Runout M.T. M.T. M.T. M.T. O.T.
	1000	75 77.5 80 85 90 95 100 105 115	41.2 42.6 44.0 46.8 46.8 49.5 52.3 55.0 57.7 63.2	33.8 34.9 36.0 38.3 40.5 42.7 45.0 47.3 51.8	$\begin{array}{c} 1.75 \times 10^{6} \\ 6.0 \times 10^{6} \\ 7.34 \times 10^{6} \\ 9.12 \times 10^{5} \\ 4.91 \times 10^{6} \\ 4.42 \times 10^{6} \\ 1.23 \times 10^{5} \\ 1.83 \times 10^{5} \\ 1.11 \times 10^{5} \\ 2.0 \times 10^{4} \end{array}$	Threads M.T. M.T. Threads M.T. M.T. M.T. M.T. M.T. 0.T.
	1200	15 20 35 60 80 90 100	8.3 11.0 19.3 33.0 44.0 49.5 55.0	6.8 9.0 15.8 27.0 36.0 40.5 45.0	$\begin{array}{c} 1.0 \times 10^{7} \\ 1.78 \times 10^{6} \\ 3.95 \times 10^{5} \\ 2.4 \times 10^{4} \\ 8 \times 10^{3} \\ 4 \times 10^{3} \\ 3 \times 10^{3} \end{array}$	Runout M.T. M.T. M.T. M.T. M.T. M.T.
Electroless Nickel Coated, Diffusion Bonded	1000	32.5 35 40 45 50 70 90	17.8 19.3 22.0 24.8 27.5 38.5 49.5	14.6 15.7 18.0 20.3 22.5 31.5 40.5	$\begin{array}{c} 3.56 \times 10^{7} \\ 1.0 \times 10^{7} \\ 2.78 \times 10^{6} \\ 5.83 \times 10^{6} \\ 9.78 \times 10^{5} \\ 8.0 \times 10^{4} \\ 8.0 \times 10^{3} \end{array}$	M.T. Runout M.T. M.T. M.T. M.T. O.T.

Table 1. FATIGUE TEST RESULTS ON Ti-6A1-2Sn-4Zr-6Mo SPECIMEN

\*Runout: In excess of 107 cycles M.T. = Middle Third O.T. = Outer Third

The microstructural changes occurring with time, at temperature, are shown in Figures 3a, b, c, and 4. The changes are caused by the combination of stress and time at temperature, which in turn affects the life of the material. The resulting microstructures in all cases are very fine; therefore the photographs were prepared at a magnification of 2100 times. Comparing structures of fatigued specimens with the structure in the as-received condition (Figure 5) the following observations are made. In the as-received condition, the microstructure consists of alpha (light) grains in the form of various sized rods and plates in a transformed beta matrix. In the longitudinal direction, the alpha appears primarily as curved rods with a mixture of globular phase. This is obviously the result of sectioning through the aligned plates and rods observed in the longitudinal sections. The microstructures of the fatigued specimens illustrate the rapid refinement of the elongated as-received structure during testing. The elongated grains are being transformed, under the action of time at temperature, to equiaxed smaller grains.



Figure 2. Axial load fatigue for unnotched Ti-6AI-2Sn-4Zr-6Mo.

The fracture surface contains a topographical record of the events of crack propagation. A schematic of the fracture surface viewed at low magnification is shown in Figure 6. A general system of classification of fracture surfaces, based upon their macroscopic configuration, includes markings that are either parallel or perpendicular to the direction of crack propagation or may include areas that are fibrous, granular, or cleaved in appearance.<sup>5</sup> A macrophotograph of the fractured surface of a specimen tested at 900 F is shown, along with the electron micrographs, in Figure 7. The microphotograph depicts three distinctive fracture zones. The area propagating from the origin of fracture appears radial and was more discolored, a deep yellow, than the other fracture surfaces, giving evidence that this fracture surface was formed early and exposed to the high temperature environment longer. The stress increased with the reduction in cross-sectional area leading to the radial fibrous zone. The final zone in the fracturing process is the shear lip.

Transmission electron fractographs of each fracture surface are shown in Figure 7. The fracture surface in the area of the origin appears to be composed of a mixed mode of quasi-cleavage and intergranular fracture. The area behind the origin appears to be principally dimpled while the shear lip areas appear also to be composed of dimples, some of which have been stretched.

5. BUNSHAH, R. F., ed. Techniques for the Direct Observation of Structure and Imperfections. Techniques of Metals Research, v. II, part I, Interscience Publishers, New York, 1968.







Figure 4. Axial load fatigue for coated Ti-6Al-2Sn-4Zr-6Mo, diffusionbonded electroless nickel at 1000 F.

#### DISCUSSION

The required microstructure for the alloy Ti-6Al-2Sn-4Zr-6Mo which results in an optimum combination of both fracture toughness and tensile ductility at yield strengths of 170 to 180 ksi has been identified.<sup>6</sup> Such a structure contains primarily a matrix of coarse acicular alpha and aged beta.<sup>6</sup> When the alpha particles are irregular in shape and somewhat elongated, a crack propagating in any plane not parallel to the axial face will find a more difficult path offered than when the microstructure consists of more equiaxed alpha. The microstructure of the longitudinal section in Figure 5 consists of a mixture of various sized rods and plates of alpha elongated in the direction of rolling (bar axis). Many titanium alloys are placed in service in the annealed condition. The annealing of titanium alloys serves primarily to provide adequate toughness and maximum ductility at room temperature and dimensional and structural stability at elevated temperatures. The material, therefore, was tested in the as-received annealed condition and the microstructure, consisting of elongated alpha particles in the rolling direction and perpendicular to direction of a propagating crack, offers resistance to fatigue crack propagation.

The microstructural changes occurring at 900 F during fatigue testing do not affect the fatigue life of this material as can be seen when comparing the curves for 700 F, 400 F, and RT (Figure 2). The greatest change in fatigue life occurs at 1200 F and the accompanying microstructural change (Figure 3c) at  $10^6$  cycles is to a finer structure with less defined alpha grain boundaries. The microstructural changes which occurred at lower temperatures are less dramatic; however, it is believed that a similar refinement of structure is taking place.

6. HALL, J. A., PIERCE, C. M., RUCKLE, D. L., and SPRAGUE, R. A. Properties Microstructure Relationships in Titanium Alloy Ti-6Al-2Sn-4Zr-6Mo. AFML-TR-71-206, November 1971.



Mag. 2500X

Mag. 500X

a. Ti-6AI-2Sn-4Zr-6Mo, as-received, sectioned parallel to rod axis.



Mag. 1000X

Mag. 500X

b. Ti-6AI-2Sn-4Zr-6Mo, as-received, sectioned transverse to rod axis.

Figure 5. Microstructure of Ti-6AI-2Sn-4Zr-6Mo. Etchant, 5 ml HCl, 500 ml H<sub>2</sub>O, 10 ml Zefferin chloride, 1 ml HF.





Figure 6. Schematic of the fracture surface zones on round tensile fatigue specimens.

microstructural changes for 900 F and 1000 F are shown in Figures 3a and 6 and consist of refined alpha in a beta matrix. Photographs of the coated specimens tested at 1000 F (Figure 4) do not indicate an unusual influence of the coating on the refinement of the microstructure compared to uncoated specimens as would be expected. Grain refinement appears to offer an improved path for a propagating crack.

Mechanical twinning can become an important mode of deformation in closepacked hexagonal metals during high stress fatigue.<sup>7</sup> A characteristic of twinned regions in a fatigue specimen is the presence of small kink boundaries. The complexity and fineness of the as-received structure made microstructural examination difficult. Following testing, this microstructure was further refined, therefore neither kink nor twin regions could be observed in the area of the fracture.

The use of heat-resistant alloys possessing high stability contributes to an increase in the life of gas turbine engines. A further increase is connected with certain structural and technological measures, one of which is the use of heat-resistant coatings to protect the surface of components from oxidation and erosion. It has been shown that the effect of heat-resistant coatings on the fatigue life of heat-resistant materials is complex in character. The technique of depositing the coating, the properties of the basic material, etc., all influence the effect the coating will have on fatigue life.<sup>8</sup>

- 7. PARTRIDGE, P. G. Cyclic Twinning in Fatigued Close-Packed Hexagonal Metals. The Philosophical Magazine, v. XII, November 1965.
- 8. GELL, M., and LEVERANT, G. R. Mechanisms of High Temperature Fatigue in Fatigue at Elevated Temperatures. American Society for Testing and Materials, Special Technical Publication No. 520, 1973, p. 37.





Figure 7. Macrograph (Mag. 10X) and electron micrographs (Mag. 3000X and 6500X) of Ti-6AI-2Sn-4Zr-6Mo tested at 900 F, load 105 ksi, lifetime of 5.8 x 10<sup>6</sup> cycles.

The application of a coating can have a number of relevant effects on the fatigue properties of the coating-substrate composite: (1) the deformation behavior of the substrate may be changed because of the presence of a surface layer having a different elastic modulus and yield strength from that of the substrate; (2) as long as the coating is sound, oxygen is kept away from the substrate and the effects of the oxygen absorption and gross oxidation are eliminated; and (3) the fatigue properties of the coating in a gaseous environment become important. If the fatigue properties of the coating are better than that of the substrate. increased life may be expected. On the other hand, if the fatigue properties of the coating are poorer than the substrate, cracks in the coating will serve as surface notches and as paths for the oxygen to reach the substrate. Reduced fatigue life of the composite would then be expected.<sup>8</sup> Figure 8 shows cracking initiated at the surface of the coating. The photomicrographs are of the surface (cross section) near the fracture. It can be seen that the cracks extend considerably into the substrate. The coating is obviously more brittle than the substrate. These cracks in the coating serve as surface notches lowering the fatigue resistance of the composite structure.

Voids are usually formed at grain boundaries. These voids will tend to grow because of the triaxial stresses and then coalesce or join together by rupturing during crack growth, resulting in a surface that is fibered in appearance. Fracture initiation is usually observed macroscopically as a fibrous zone, which represents slow crack growth and propagation. It then changes to rapid or unstable propagation as shown in the radial zone. The final zone of the fracturing process usually is the formation of the shear lip, the surface of which is smooth and tends to be inclined 45° to the tensile axis.

The initiation zone observed in macroscopic examination of the fracture surface is a radial zone. The markings are parallel to the direction of crack propagation and hence assume a spoke or star-shaped appearance. Radial markings usually result from either cleavage or low-energy tear-type propagation.<sup>9</sup> Electron micrographs at the origin of fracture indicate the failure mechanism occurred by quasi-cleavage and intergranular fracture. The characteristic feature of quasicleavage is the high density of short curved lines which results from the formation of ridges when cracks grow together within the facet. Quasi-cleavage is a mixed mechanism involving both microvoid coalescence and cleavage.<sup>9,10</sup> In addition, there appears to be grain boundary separation. Therefore, the mechanism in this zone appears to consist of a combination of microvoid coalescence, cleavage, and intergranular fracture.

In the fibrous zone, failure usually occurs by normal rupture. On a microscopic scale, the fracture is quite jagged since the crack advances by shear fracture (void coalescence) on alternating planes inclined at 40° to 50° to the tensile axis.<sup>9</sup> This form of fracture is commonly labeled normal rupture when the fracturing path is normal to the tensile axis or fibrous when the jagged surface has fibrous or silky appearance. The appearance of the second zone in Figure 7 is radial fibrous and appears to be a result of dimpled fracture.

TETELMAN, A. S., and MCEVILY, A. J., Jr. Fracture of Structural Materials. John Wiley and Sons, Inc., New York, 1967.
 Interpretation of Transmission-Electron-Microscope Fractograph. American Society for Metals, Metals Handbook, v. 9, 1974, p. 79-92.



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Figure 8. Ti-6Al-2Sn-4Zr-6Mo, coated with diffusion-bonded electroless nickel tested at 1000 F, 45 ksi stress, life of 5.8 x 10<sup>6</sup> cycles.

The final zone of the fracturing process is the formation of the shear lip which is converted from the radial fibrous zone. The entire area of the remaining cross section is probably plastically flowing as the shear lip develops in the plastic zone. Fracture is rapid and occurs by shear rupture. The fracture surface is extremely smooth and tends to be inclined at 45° to the tensile axis. The electron micrographs show a dimpled structure, some of which have been stretched. Features of shear rupture are dimples stretched out in the shearing direction.<sup>8</sup>

The sequence of zones of the fracture path is changed from that which is usually observed on fracture surfaces tested at room temperature. This may be due to the fact that testing in this study was conducted at elevated temperatures.

## CONCLUSIONS

1. Axial load fatigue data of unnotched Ti-6Al-2Sn-4Zr-6Mo revealed no change in fatigue strength at 900 F compared to room temperature, 400 F, and 700 F. At 1000 F the decrease in fatigue strength from that at 900 F and below becomes noticeable. At 1200 F the decrease is significant.

2. The fatigue strength-life curve of diffusion-bonded electroless nickelcoated specimens tested at 1000 F falls midway between results of uncoated specimens tested at 1000 F and 1200 F. The reduction of fatigue life may be attributed to the notch effect of cracks observed in the plating, a result of the poorer fatigue properties of the coating. There are other erosion-resistant coatings which are less deleterious to fatigue properties.<sup>1</sup> The effect of these coatings on elevated temperature fatigue properties of titanium alloys should be determined.

3. Microstructural changes occurring with time, at temperature, indicate refinement of structure with time and temperature.

4. Macrofractographs reveal three distinct areas: an initial radial area, followed by a radial fibrous, and finally a shear lip.

5. Transmission electron microscopy of surfaces fractured at 900 F indicate a mixed mode at the origin of quasi-cleavage and intergranular fracture while the second area (radial fibrous) appears to be principally dimpled. The shear lip occurs by shear fracture.

#### ACKNOWLEDGMENT

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