NASA TECHNICAL NOTE



NASA TN D-8214



LOAN COPY: RETURN TO AFWL TECHNICAL LIBRAR', KIRTLAND AFB, N. M.

SELECTED FRETTING-WEAR-RESISTANT COATINGS FOR TITANIUM - 6-PERCENT-ALUMINUM -4-PERCENT-VANADIUM ALLOY

Robert C. Bill Lewis Research Center Cleveland, Ohio 44135





NATIONAL AERONAUTICS AND SPACE ADMINISTRATION . WASHINGTON, D. C. . APRIL 1976

				0733773	
1. Report No.	2. Government Access	ion No.	3. Recipient's Catalog	No.	
NASA TN D-8214			5. Report Date	· 	
4. Title and Subtitle SELECTED FRETTING-WEAR-RESISTANT COATINGS FOR			April 1976		
TITANIUM - 6-PERCENT-ALUMINUM - 4-PERCENT-			6. Performing Organiz	ation Code	
VANADIUM ALLOY					
7. Author(s)			8. Performing Organization Report No.		
Robert C. Bill			E-8594		
O. Parforming Occanization Name and Address			10, Work Unit No.		
9. Performing Organization Name and Address Lowing Page and Contant			505-04		
Lewis Research Center		11. Contract or Grant No.			
National Aeronautics and Space	<u> </u>				
Cleveland, Ohio 44135 12. Sponsoring Agency Name and Address			13. Type of Report and Period Covered		
National Aeronautics and Space Administration			Technical Note		
Washington, D.C. 20546		14. Sponsoring Agency	Code		
15. Supplementary Notes					
16. Abstract					
A titanium - 6-percent-alumi	num - 4-percent-v	anadium alloy (Ti-	6Al-4V) was sub	jected to	
fretting-wear exposures again	nst uncoated Ti-6A	l-4V as a baseline	and against vari	ous coatings	
and surface treatments applied to Ti-6Al-4V. The coatings evaluated included plasma-sprayed					
tungsten carbide with 12 percent cobalt, aluminum oxide with 13 percent titanium oxide, chrom-					
ium oxide, and aluminum bronze with 10 percent aromatic polyester; polymer-bonded polyimide,					
	polyimide with graphite fluoride, polyimide with molybdenum disulfide (MoS ₂), and methyl phenyl				
silicone bonded MoS ₂ ; a preoxidation surface treatment, a nitride surface treatment, and a sput-					
tered MoS ₂ coating. Results of wear measurements on both the coated and uncoated surfaces					
_					
after 3×10^5 fretting cycles indicated that the polyimide coating was the most wear resistant and					
caused the least wear to the uncoated mating surface.					
		lao ni ilian			
17. Key Words (Suggested by Author(s))		18. Distribution Statement			
-	oatings	Unclassified - unlimited			
	Fretting wear Surface treatments		STAR Category 26		
Titanium alloy					
10. Committee Cloself (of able to	100 6		21 No. 15	22. Price*	
19. Security Classif. (of this report)	20. Security Classif. (c		21. No. of Pages		
Unclassified	Uncla	assified	24	\$3.25	

^{*} For sale by the National Technical Information Service, Springfield, Virginia 22161

SELECTED FRETTING-WEAR-RESISTANT COATINGS FOR TITANIUM - 6-PERCENT-ALUMINUM - 4-PERCENT-VANADIUM ALLOY

by Robert C. Bill

Lewis Research Center

SUMMARY

A titanium - 6-percent-aluminum - 4-percent-vanadium alloy (Ti-6Al-4V), in the solution-treated overaged condition, was subjected to fretting exposures against uncoated Ti-6Al-4V as a baseline and against various coatings and surface treatments applied to Ti-6Al-4V. Fretting experiments were conducted in dry air and in saturated air by using a 4.7-millimeter-radius hemisphere on a flat surface. Typical conditions included a 1.47-newton normal load, an 80-hertz frequency, and a 70-micrometer amplitude at room temperature. The coatings evaluated included plasma-sprayed tungsten carbide with 12 percent cobalt, aluminum oxide $(\mathrm{Al_2O_3})$ with 13 percent titanium oxide (TiO2), chromium oxide, and aluminum bronze with 10 percent aromatic polyester; polymer-bonded polyimide, polyimide with graphite fluoride, polyimide with molybdenum disulfide (MoS₂), and methyl phenyl silicone bonded MoS₂; a preoxidation surface treatment, a nitride surface treatment; and a sputtered MoS2 coating. Results of wear measurements on both the coated and uncoated surfaces after 3×10⁵ cycles indicated that the polyimide coating was the most wear resistant and caused the least wear to the uncoated surfaces. Increased exposure, however, showed a sharp increase in fretting wear rate after 10^6 cycles. Surface nitrided Ti-6Al-4V fretted against a polytetrafluoroethylene impregnated $\mathrm{Al_2O_3}\text{-TiO_2}$ coating on Ti-6Al-4V resulted in immeasurable wear to both surfaces.

INTRODUCTION

Bolted interfaces, pinned joints, spline couplings, and gripped components are part of many aircraft structural and engine assemblies. Vibrations, fluctuating major loads, and misalinements between rotating components, inherent in any aircraft, promote small amounts of oscillatory slip between mating surfaces with the result that fretting is a potential problem at all interfaces. There are two significant consequences of fretting.

First, the loss of specified interfacial fits or clearances due to fretting wear can lead to increases in vibrational amplitudes and ultimate component failure, possibly at a location remote from the site of the fretting wear. Second, there is the possibility of direct reduction in fatigue strength of components in an assembly due to fretting action at the interfaces. The fatigue strength reduction arises because fretting wear promotes crack initiation at the mating surfaces. Depending on the stress state of the fretted components, the cracks initiated may propagate to cause early failure.

An important material used in many airframe and engine applications is the titanium - 6-percent-aluminum - 4-percent-vanadium alloy (Ti-6Al-4V). Usually this alloy is used in a hardened condition, in which it is prone to accelerated fatigue failure due to surface flaws. The effect of fretting on the fatigue strength of Ti-6Al-4V was studied (refs. 1 and 2), and in general fretting severely reduced the fatigue life.

The purpose of this report is to establish the fretting-wear behavior of Ti-6Al-4V and to evaluate various coatings as fretting wear inhibitors for this alloy. Plasmasprayed coatings, polymer-bonded coatings, and some surface treatments were examined. Experimental combinations consisted of an uncoated Ti-6Al-4V hemisphere fretted against a coated or treated flat surface. The plasma-sprayed coatings included cobalt (Co)-bonded tungsten carbide (WC), an aluminum oxide (Al $_2$ O $_3$) - titanium oxide (TiO $_2$) composite, chromium oxide (Cr $_2$ O $_3$), and aluminum bronze with 10 percent aromatic polyester (Ekonol). The polymer-bonded coatings examined were 100 percent polyimide, polyimide with 60 weight percent graphite fluoride, polyimide with 75 weight percent molybdenum disulfide (MoS $_2$), and a methyl phenyl silicone bonded MoS $_2$ with antimony trioxide (Sb $_2$ O $_3$). The surface treatments that were evaluated included preoxidation, surface nitride formation, and sputter-coated MoS $_2$.

Room-temperature dry air and saturated air and varied load, fretting exposure (number of cycles), and temperature were used in evaluating the material combinations. Fretting-wear measurements made on both the coated or treated surface and the uncoated surface were the primary means of evaluation. In addition, microscopic examination of fretting surface damage was conducted.

APPARATUS

A schematic diagram of the fretting rig is shown in figure 1, and a detailed description of its operation is included in reference 3. In essence, a linear oscillatory motion is provided by an electromagnetically driven vibrator with the frequency controlled by a variable oscillator. The load is applied to the specimens by placing precision weights on a pan which is hung from the load arm.

The fretting specimens consist of an upper, sationary, 4.76-millimeter-radius, hemispherical tip in contact with a lower flat surface which is driven by the vibrator.

During high-temperature experiments, the specimens and grip assemblies were surrounded by a 310 stainless steel susceptor which was heated by an induction coil. The temperature was monitored by a thermocouple probe mounted in the lower grip.

A dry air environment was provided by flowing air through an absorption drier and then into the experimental chamber. In this way moisture content was kept in the range 10 to 100 parts per million. When a moisture-saturated environment was desired, the air was bubbled through a water-filled column and then blowed into the chamber.

PROCEDURE

The preparation of the specimen surfaces before a fretting experiment depended on the type of coating or surface treatment applied to the surface.

The bare Ti-6Al-4V surfaces were hand lapped with levigated alumina and then washed in tap water with a polishing cloth to remove the alumina. Following the washing the specimens were rinsed in absolute ethanol, rinsed in distilled water, and then set aside to dry.

The flat plasma-sprayed surfaces were machine lapped, with approximately 75 to 105 micrometers (3 to 5 mils) of coating being removed in the process. In this way the very rough abrasive as-sprayed coating was smoothed so that, disregarding the surface pores, the root-mean-square roughness of the coating was about 0.5 micrometer (20 μ in.). The lapped surface was then ultrasonically cleaned in absolute ethanol, rinsed in ethanol, rinsed in distilled water, and allowed to dry.

The surfaces with polymer-bonded coatings and those with the various surface treatments were washed in tap water, rinsed in ethanol, and rinsed in distilled water before being subjected to fretting.

Following the surface preparation treatment, the specimens were assembled into the grips according to the desired combination, but always with the uncoated Ti-6Al-4V hemisphere in the upper grip and either a coated or an uncoated Ti-6Al-4V flat surface in the lower grip. The test chamber was then purged with the selected atmosphere (dry air or saturated air) for 15 minutes. If the experiment was to be conducted at elevated temperature (150° or 300° C), the heating period was simultaneous with the purge.

The fretting exposure was initiated by adding the required weight to the load pan to bring the contact force to the desired level, usually 1.47 newtons. The amplitude of the fretting motion was 70 micrometers (0.00275 in.), and the frequency of the fretting motion was 80 ± 0.2 hertz. The standard duration of the fretting exposure was 3×10^5 cycles (1 hr).

Following each fretting experiment, the fretting scars on both surfaces were photomicrographed to record the size and features of the wear scars and the debris accumulation. The loose debris was then rinsed off with ethyl alcohol, and a light-section

microscope was used to measure the maximum depth and diameter of the wear scars on both surfaces. Wear volumes were calculated by applying spherical cap approximations to the wear scar geometry.

In principle, the light-section measurement technique consists of directing a plane beam of light obliquely (with a 45° angle of incidence) at the specimen surface. The reflected light beam is viewed through an optical microscope. If a plane surface is viewed, the light beam appears as a straight line across the field of view; if a hemispherical surface is viewed, the light beam appears as a smooth curve. Wear or surface distress due to fretting action shows up as deviations from the smooth profile of the surrounding surface. With the surrounding surface profile used as the datum, wear depth measurements may be made by means of a built-in crosshair system. The diameter of the wear area may be measured by manipulation of an indexed micrometer stage translation. Wear depth measurements are accurate to within 0.5 micrometer, and diameter measurements are accurate to about 20 micrometers. The smallest wear scars examined in this investigation had a nominal depth of about 0.5 micrometer and a diameter of roughly 80 micrometers. Thus, these wear scars represented the practical limit of resolution for the light-section technique, with an uncertainty in wear volume resulting from measurement of about 50 percent. The wear volume uncertainty due to measurement techniques for the typical wear scars in this investigation was of the order of 10 percent.

MATERIALS

The Ti-6Al-4V used in this investigation was in the solution-treated overaged condition, so that the Rockwell-B hardness was 108.

The plasma-sprayed coatings, Co-bonded WC, Al_2O_3 with TiO_2 , Cr_2O_3 , and aluminum bronze, were all applied to an initial thickness of 250 micrometers (0.010 in.); they had an estimated density, based on the experience of the supplier, of over 95 percent. The Co-bonded WC consisted of 12 weight percent Co binder; the Al_2O_3 - TiO_2 composite contained 13 weight percent TiO_2 ; and the aluminum bronze coating was a copper - 10-percent-aluminum bronze with 10 percent by volume of an aromatic polyester cosprayed with the aluminum bronze. With the exception of the aluminum bronze, the plasma-sprayed coatings are characterized as hard, protective, wear-resistant coatings.

Solution-sprayed surface coatings of polyimide without additives, polyimide with 60 weight percent graphite fluoride, and polyimide with 75 weight percent MoS₂ were evaluated. The application procedure is described in detail in reference 4. Briefly, the coatings were applied to a final thickness of about 20 micrometers, baked for 1 hour at 100° C to volatilize the thinner, and then baked for 1 hour at 300° C to cure the polymer. The choice of polyimide as a binder was based on its combination of mechanical

strength and low friction and wear (ref. 5). Graphite fluoride was investigated as a solid lubricant and was very effective in a polyimide binder under unidirectional sliding conditions (refs. 4 and 6).

The sprayed methyl phenyl silicone bonded MoS_2 film was originally formulated as a wear-resistant coating requiring no elevated temperature curing (ref. 7). In addition to MoS_2 , the film included an $\operatorname{Sb}_2\operatorname{O}_3$ additive as an oxidation stabilizer. The methyl phenyl silicone bonded film has been an effective wear-resistant coating for titanium alloys.

The preoxidation treatment consisted of heating the Ti-6Al-4V in room air to 650° C for $1\frac{1}{2}$ hours. No softening of the alloy accompanied the preoxidation, as the overaging treatment had been carried out at 700° C. The oxide film formed was measured to be 2 micrometers thick.

The nitride layer was applied to the surface by means of a vapor diffusion process in which ammonia was the active gas. The treatment temperature was approximately 540° C, and the process lasted about 24 hours. Since the nitride layer was diffused, the thickness was difficult to specify. After treatment, the nitrided surface exhibited a bright yellow color.

The sputtered ${\rm MoS}_2$ coating was deposited by a radiofrequency sputtering process, the details of which are described in reference 8. The advantages of radiofrequency sputtering as a deposition process include microstructural control, tenacious bonding, and the attainment of nearly theoretical density. The coating applied was between 0.4 and 0.5 micrometer thick.

RESULTS AND DISCUSSION

Titanium-Aluminum-Vanadium Alloy

Figure 2 shows the wear volume as a function of the number of cycles for uncoated Ti-6Al-4V fretted against uncoated Ti-6Al-4V. An average curve drawn through the values for wear volume has a shape similar to that for high-purity titanium in reference 9. There is an initial period of high fretting-wear rate followed by a plateau, referred to as a conditioning period by Hurricks (ref. 10). The plateau in figure 2 is less pronounced than the one in reference 9. Finally, the wear rate increases again after 3×10^5 to 10^6 cycles.

The conditioning period is described in reference 10 as being a period of mechanical fatigue in the work-hardened contact zone. This description is supported by microscopic examinations made in the study of reference 9, in which fatigue-initiated spall pits were first observed during the conditioning part of the fretting exposure. The final increased wear rate, or steady-state wear as it is called in the literature, is currently believed to be the result of corrosion-assisted disintegration of the heavily fatigued con-

tact zone (refs. 10 to 12).

The sequency of photographs in figure 3 shows that the final increase in wear rate is accompanied by the generation of large amounts of debris. The effect is consistent with the observation made in the study of reference 9, that debris particles originated from the disintegration of the edges of fatigue spall pits. Such spall pits may be seen in figure 3(d), taken after 3×10^5 fretting cycles. The generation of spall pits proceeds by crack initiation and propagation. The implication is that the fretting process provides a mechanism of direct crack initiation which can lead to accelerated fatigue failures under adverse stress conditions.

A major difference between the fretting behavior of high-purity titanium (ref. 9) and that of Ti-6Al-4V is the number of cycles required before the final onset of steady-state wear occurred. For pure titanium 3×10^4 cycles were required, and for the alloy 3×10^5 cycles. This difference can be accounted for by the greater mechanical strength of the alloy. A larger number of fretting cycles was required to develop the fatigue damage that set the stage for debris generation and increased wear.

The effect of normal load on the fretting wear of Ti-6Al-4V was also investigated. No measurable wear was observed after 3×10^5 cycles under 0.74- and 0.29-newton loads. The wear under a 1.47-newton load after 3×10^5 cycles was 10×10^{-5} to 20×10^{-5} cubic millimeter. Under a 2.94-newton load seizure occurred, and a meaningful wear measurement could not be made.

After fretting exposures of 3×10^5 cycles at temperatures of 150^0 and 300^0 C, the wear volumes were, respectively, 2 and 2.5 times greater than that resulting from room-temperature fretting. This trend is in general agreement with that observed for high-purity titanium up to about 500^0 C (ref. 13). The effect is thought to be caused by an increased rate of breakdown of the wear scar surface under the influence of the more reactive (higher temperature) environment. At temperatures up to at least 300^0 C, the thin oxide film that forms on Ti-6Al-4V is not effective in reducing wear.

Plasma-Sprayed Coatings

From the standpoint of minimizing damage to the opposing uncoated Ti-6Al-4V surface, the aluminum bronze with the aromatic polyester was the most effective of the plasma-sprayed coatings (fig. 4(a)). The sprayed aluminum bronze and aromatic polyester may be considered a sacrificial coating because most of the wear occurred to the coating itself rather than to the opposing uncoated Ti-6Al-4V surface. As such, the coating should be applied to the surface opposing the wear-critical or fatigue-critical surface.

The remaining three plasma-sprayed coatings, WC-Co, Al_2O_3 -TiO₂, and Cr_2O_3 , may be considered hard, protective, wear-resistant coatings. Coatings such as these

should be applied to the wear-critical or fatigue-critical surface, but with some caution. It is often observed that a significant reduction in the fatigue strength of the substrate material accompanies the application of a hard coating even without the incidence of fretting or other surface distress (ref. 14). This effect must be weighed against the reduction in fatigue strength associated with surface distress caused by fretting (refs. 1 and 2). A decision may then be made as to whether a fretting-wear-resistant coating would be beneficial in a fretting-fatigue situation.

The most wear resistant of the hard coatings was the ${\rm Al_2O_3\text{-}TiO_2}$ (fig. 4(b)). It was also the least sensitive of the hard plasma-sprayed materials to the presence of moisture. The maximum wear scar depth on the coating was measured to be 3 micrometers under dry air conditions and 1 micrometer under moisture-saturated conditions. The wear scars resulting from fretting ${\rm Al_2O_3\text{-}TiO_2\text{-}coated}$ Ti-6Al-4V against uncoated Ti-6Al-4V are shown in figure 5. Under both dry air and moisture-saturated air conditions, two-shaded gray fretting debris was generated, as happened when Ti-6Al-4V was fretted against Ti-6Al-4V. The bulk of the debris was thus considered to have come from the Ti-6Al-4V surface.

As may be seen in figure 4(a), the $\mathrm{Al_2O_3}\text{-TiO_2}$ coating produced the least fretting wear volume of the hard plasma-sprayed coatings on the unprotected Ti-6Al-4V surface. The wear scar on the uncoated Ti-6Al-4V surface after fretting against the $\mathrm{Al_2O_3}\text{-TiO_2}$ coating is shown in the light-section photograph of figure 6(a). There is evidence of pitting and surface roughening due to abrasive interaction with the hard coating.

In comparison, the wear scar resulting from fretting against the WC-Co coating, shown in figure 6(b), is much smoother. This difference is believed to be the result of two effects associated with the cobalt binder in the WC-Co. First, the cobalt has been observed under unidirectional sliding conditions to mitigate the abrasive action of hard ceramic particles by forming a thin metallic film (ref. 15). Second, the cobalt binder would be expected to show that cobalt has associated with it properties of low adhesion (ref. 16). Together these two effects would reduce the amount of abrasive wear and prevent adhesive damage and metal transfer.

The performance of the ${\rm Al_2O_3\text{-}TiO_2}$ coating was improved from the standpoints of wear and fretting-induced surface distress when the coating was impregnated with Teflon (fig. 6(c)). When the mating Ti-6Al-4V surface was subjected to a nitride treatment, the wear to the ${\rm Al_2O_3\text{-}TiO_2\text{-}coated}$ surface and the wear to the nitrided surface were both immeasurable (figs. 6(d) and (e)). The nitride seemed to be intact over about 70 percent of the fretted surface.

Polymer-Bonded Coatings

In comparison with the plasma-sprayed coatings, the polymer-bonded coatings are

soft, low-modulus materials. They would therefore be expected to work most effectively when applied as wear buffer or self-lubricating coatings. The most significant wear measurement would then be the wear to the uncoated mating surface. Wear to the coatings themselves would be indicative of the relative coating lives.

Figure 7 summarizes the performance of the polymer-bonded films. The wear measurements made on the uncoated surfaces (fig. 7(a)), with bare Ti-6Al-4V against bare Ti-6Al-4V as a baseline, show that a significant reduction in fretting wear was realized for all the films. For the polyimide-bonded films, no improvement over the straight polyimide resulted when the solid lubricants graphite fluoride and molybdenum disulfide were added to the films. The presence of moisture increased the wear of the uncoated surface by a factor of 2 to 3 for the polyimide-bonded films, except for the film with the ${\rm MoS}_2$ additive, which reduced wear by a factor of about 3. It would thus seem that the ${\rm MoS}_2$ in the film somehow mitigated the adverse effect that moisture would have on the fretting of the titanium alloy, perhaps by acting as a moisture getter at the interface.

The wear to the polymer-bonded films is summarized in figure 7(b). The most significant observation is that the wear of the polyimide films increased by a factor of about 3 in dry air and 2 in moist air with the addition of solid lubricant additives. The wear of the silicone-bonded film was about two orders of magnitude higher than that of the polyimide films in dry air, but in moist air the difference in wear was less than an order of magnitude.

Figure 8 shows the fretting wear of the polymer-bonded films as a function of fretting exposure. The most apparent feature of the polyimide-bonded films is the increasing wear rate with extended exposure, especially for films with additives. This is suggestive of a fatigue mechanism exacerbated by the presence of discrete lubricant particles. Even after 10⁶ cycles, none of the polyimide-bonded films had been penetrated. A thin, smeared film of polyimide, with distinct lenticular-shaped deformation features, shown in figure 9, remained on the Ti-6Al-4V surface.

The methyl phenyl silicone bonded MoS_2 film wore at a uniform rate up to about 3×10^5 cycles, as may be seen in figure 8. Beyond this exposure the apparent coating wear rate decreased significantly. Microscopic examination revealed, however, that film penetration had occurred after some number of cycles between 10^5 and 10^6 . Figure 10 reveals the exposure of the Ti-6Al-4V substrate after 10^6 cycles, with the generation of characteristic oxidized debris.

A direct comparison of the polyimide-bonded films with the methyl phenyl silicone bonded film is difficult. The polyimide coatings were more durable in both dry and saturated air, but since the coating wear mechanism for all the polymer-bonded films involved plastic flow of the coating under the concentrated contact, the relative coating wear rate differences might be less significant under conforming contact applications, in which the flow would be constrained. Also, the relative influence of moisture on the

two types of coatings should be considered. Perhaps with prolonged fretting exposure ($> 3 \times 10^5$ cycles) in moist air, the wear of the polyimide- and silicone-bonded coatings would be similar. A final important consideration for certain applications is the fact that the methyl phenyl silicone bonded film was cured at room temperature, while the polyimide-bonded film application involved a high-temperature cure.

Preoxidation, Nitride, and Sputtered Molybdenum Disulfide Surface Treatments

As may be seen in figure 11, a slight increase in the amount of wear to the untreated Ti-6Al-4V hemisphere occurred when it was fretted against preoxidized Ti-6Al-4V in dry air. Transferred material was observed on the preoxidized specimen surface.

When fretting was conducted in a moisture-saturated environment, the preoxidation treatment resulted in a very large reduction in wear to the untreated hemisphere. Furthermore, there was a large reduction in the amount of material transferred to the preoxidized surface.

Figure 12 shows the wear volume measured on untreated Ti-6Al-4V as a function of the number of fretting cycles against preoxidized Ti-6Al-4V. As a baseline, the wear volumes from figure 4 are also shown. The baseline wear rate after about 10^5 cycles was higher than the wear rate for the untreated surface against the preoxidized surface. Thus, even in dry air some reduction in wear to the untreated surface might result from preoxidizing the Ti-6Al-4V mating surface if fretting exposures are prolonged (more than 10^6 cycles). The maximum wear to the preoxidized hemisphere after 10^6 cycles in dry air was only about 10^{-4} cubic millimeter.

The results obtained for the nitride surface treatment were similar to those for the preoxidized treatment. In both cases, the wear to the untreated specimen was greater in dry air than in moisture-saturated air, and the magnitudes of the wear volumes were about the same.

The sputtered ${\rm MoS}_2$ film effectively eliminated fretting damage to the uncoated mating surface in dry air for exposures of 3×10^5 cycles (fig. 11). The coated specimen also suffered relatively mild damage in dry air with no transfer from the uncoated surface observed. In moisture-saturated air, however, both the sputtered ${\rm MoS}_2$ surface and the uncoated surface showed considerable fretting damage after 3×10^5 cycles.

The features present on the wear scar surfaces of the sputtered-MoS $_2$ specimens after fretting in moist air were markedly different from those present after fretting in dry air. After 3×10^5 cycles in moist air, the fretted surface (fig. 13(a)) showed all the evidence of being in the final stage of the fretting process. Specifically, the surface had the leafy or layered quality associated with the steady-state debris generation and surface cracking mechanisms of fretting. X-ray dispersion revealed no peaks that could be associated with MoS $_2$. In contrast, the fretted surface resulting from 3×10^5 cycles

in dry air (fig. 13(b)) showed very little distress. Thin sheets of material transparent to the scanning electron microscope (SEM) beam, probably sheets of MoS_2 , could be seen on the surface. The Ti-6Al-4V substrate underwent only mild damage characteristic of the very first stages of fretting wear, during which localized adhesion and deformation are the dominant mechanisms. The presence of MoS_2 was detected by X-ray dispersion analysis. Up to at least 3×10^5 cycles in dry air the sputtered MoS_2 effectively prevented fretting damage to the Ti-6Al-4V substrate.

Progressive fretting exposure experiments indicated that in dry air the wear volumes on both the sputtered and the untreated surfaces increased by at least an order of magnitude at some number of cycles between 3×10^5 and 10^6 . X-ray dispersion studies revealed that MoS_2 was detectable on the wear scars for both specimens after 3×10^5 cycles, but no trace was found after 10^6 cycles. Thus, the sputtered MoS_2 coating life under the imposed fretting conditions was between 3×10^5 and 10^6 cycles.

The results for all the coatings and surface treatments are summarized in figure 14. Besides the fretting wear volumes after 3×10^5 cycles, other considerations including the qualitative nature of the fretting wear damage are important. Generally though, the wear measurements summarized in figure 14 are considered to be a fair overall indication of the relative performance of the various coatings and surface treatments.

SUMMARY OF RESULTS

A study was made of selected fretting-wear-resistant coatings for a titanium - 6-percent-aluminum - 4-percent-vanadium alloy (Ti-6Al-4V). The following results were obtained:

- 1. A polyimide coating (without additives) was the best of the sacrificial or self-lubricating coatings in both dry air and moisture-saturated air.
- 2. Plasma-sprayed aluminum oxide (Al₂O₃) with 13 percent titanium oxide (TiO₂) was the best of the hard protective coatings evaluated from the standpoint of coating wear. A slight reduction in wear to the uncoated Ti-6Al-4V surface was observed with this hard coating.
- 3. When the ${\rm Al_2O_3}$ -13TiO₂ coating was impregnated with polytetrafluoroethylene and the mating surface was nitrided, wear to both surfaces was reduced to a level below the measuring technique limit.
- 4. Preoxidation and surface nitride treatment both resulted in wear-resistant surfaces. Wear to the untreated surface was reduced in moist air.

5. Sputtered molybdenum disulfide effectively reduced fretting wear to both coated and uncoated surfaces in dry air. The benefit of the sputtered coating was lost in the presence of moist air, however.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, February 24, 1976, 505-04.

REFERENCES

- 1. Hoeppner, David W.; and Goss, Gary L.: The Effect of Fretting Damage on the Fatigue Behavior of Metals. LR-25317, Lockheed-California Co. (AD-743515), 1972.
- 2. Betts, Robert K.: Wear and Fretting Fatigue Resistant Coatings for Titanium Alloys. General Electric Co. (AD-919332L; AFML-TR-74-18), 1974.
- 3. Bill, Robert C.: Fretting of Secondary-Seal-Ring Candidate Materials in Air at Temperatures to 816° C. NASA TN D-7073, 1972.
- 4. Fusaro, Robert L.; and Sliney, Harold E.: Graphite Fluoride as a Solid Lubricant in a Polyimide Biner. NASA TN D-6714, 1972.
- 5. Jones, William R. Jr., Hady, William F.; and Johnson, Robert L.: Friction and Wear of Poly (amide-imide), Polyimide, and Pyrrone Polymers at 260° C in Dry Air. NASA TN D-6353, 1971.
- 6. Fusaro, Robert L.; and Sliney, Harold E.: Preliminary Investigation of Graphite Fluoride $(CF_x)_n$ as a Solid Lubricant. NASA TN D-5097, 1969.
- 7. Benzing, Robert J.; and McConnell, Bobby D.: Wear Behavior of an Air-Drying Methyl Phenyl Silicone Bonded Molybdenum Disulfide Lubricating Film. Air Force Systems Command (AD-878234; AFML-TR-70-179), 1970.
- 8. Spalvins, Talivaldis: Lubrication With Sputtered MoS₂ Films. Am. Soc. Lub. Engrs. Trans., vol. 14, no. 4, Oct. 1971, pp. 267-274.
- 9. Bill, Robert C.: Fretting Wear in Titanium, Monel-400, and Cobalt-25 Percent Molybdenum Using Scanning Electron Microscopy. NASA TN D-6660, 1972.
- 10. Hurricks, P. L.: The Fretting Wear of Mild Steel From Room Temperature to 200° C. Wear, vol. 19, 1972, pp. 207-229.

- 11. Stowers, I. F.; and Rabinowicz, E.: The Mechanism of Fretting Wear. Lub., Tech., ASME Trans., vol. 95, Ser. F, no. 1, Jan. 1973, pp. 65-70.
- 12. Waterhouse, R. B.; and Taylor, D. E.: Fretting Debris and the Delamination Theory of Wear. Wear, vol. 29, 1974, pp. 337-344.
- 13. Bill, Robert C.: Fretting of Titanium at Temperatures to 650° C in Air. NASA TN D-8048, 1975.
- 14. Padherg, D. J.: Fretting Resistant Coatings for Titanium Alloys. McDonnell Douglas Co. (AD-914032L; AFML-TR-73-228), 1973.
- 15. Brainard, William; and Buckley, Donald H.: Dynamic SEM Studies of Tungsten Carbide Cermets. NASA TM X-71569, 1975.
- 16. Buckley, Donald H.; and Johnson, Robert L.: Marked Influence of Crystal Structure on the Friction and Wear Characteristics of Cobalt and Cobalt-Base Alloys in Vacuum to 10⁻⁹ Millimeter of Mercury. I Polycrystal and Single Crystal Cobalt. NASA TN D-2523, 1964.

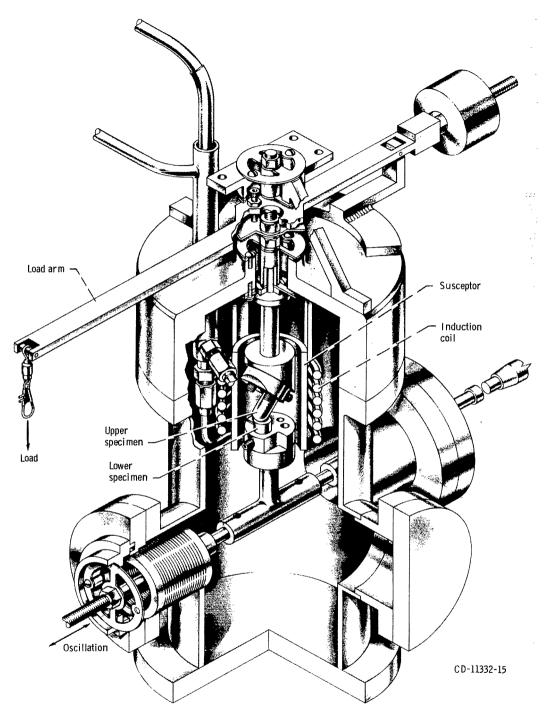


Figure 1. - Fretting apparatus.

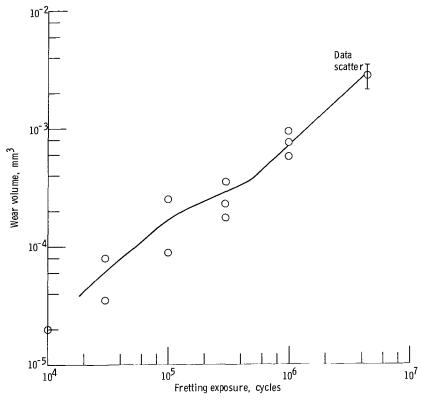


Figure 2. - Fretting wear volume as function of number of cycles for Ti-6AI-4V against Ti-6AI-4V. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; dry air; room temperature.

14

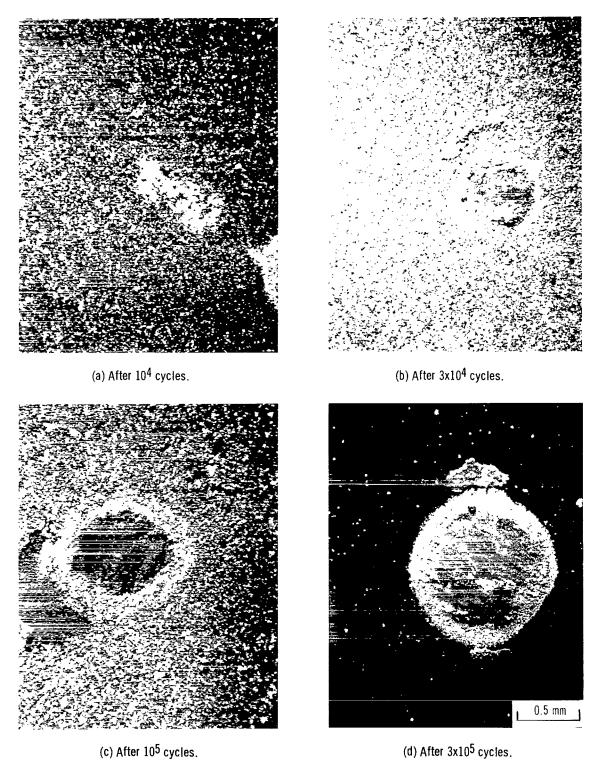


Figure 3. - Fretting wear scar sequence for Ti-6Al-4V against Ti-6Al-4V. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; dry air; room temperature.

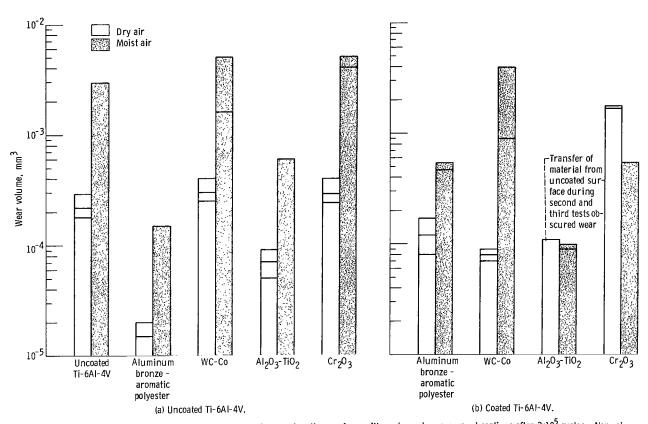


Figure 4. - Fretting wear of uncoated Ti-6Al-4V surfaces and mating surfaces with various plasma-sprayed coatings after $3x10^5$ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; room temperature.

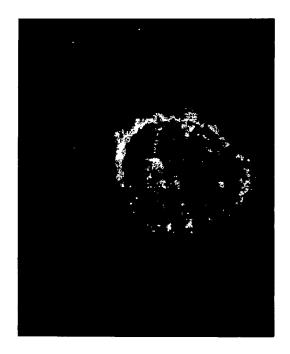


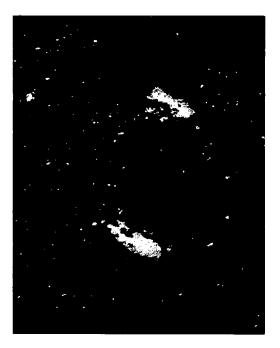
Al₂O₃-TiO₂ coated Ti-6Al-4V





(a) Moisture-saturated air.





(b) Dry air.

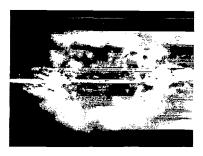
Figure 5. - Wear scars resulting from fretting uncoated Ti-6Al-4V against Al_2O_3 -13TiO2-coated Ti-6Al-4V for $3x10^5$ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; room temperature.



(a) Ti-6Al-4V (hemisphere) after fretting against Al $_2\mathrm{O}_3\text{-TiO}_2.$



(b) Ti-6Al-4V (nemisphere) after fretting against WC-Co.



(c) Ti-6A1-4V (hemisphere) after fretting against polytetrafluoroethylene-impregnated Al₂O₃-TiO₂.



(d) Polytetrafluoroethylene-impregnated Al $_2$ 03-TiO $_2$ after fretting against nitrided Ti-6Al-4V.



(e) Nitrided Ti-6Al-4V after fretting against polytetrafluoroethylene-impregnated Al $_2$ O $_3$ -TiO $_2$.

Figure 6. - Light-section surface profile micrographs of fretting wear scars after 3x10⁵ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; dry air; room temperature.

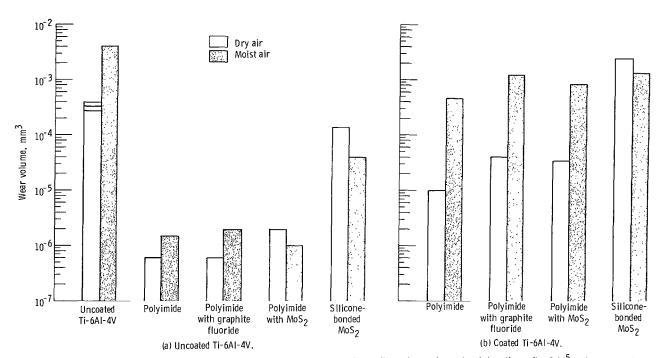


Figure 7. - Fretting wear of uncoated Ti-6Al-4V surfaces and mating surfaces with various polymer-bonded coatings after 3x10⁵ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; room temperature.

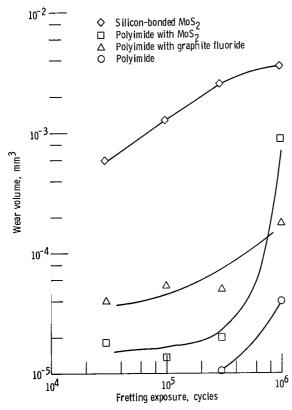


Figure 8. - Fretting wear volume as function of number of cycles for various polymer-bonded coatings against uncoated Ti-6Ai-4V. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; dry air; room temperature.

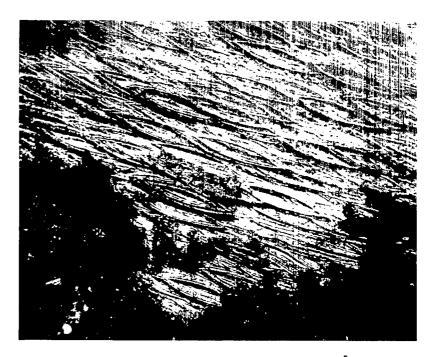


Figure 9. - Fretting wear scar on polyimde-coated Ti-6AI-4V after 3x10⁵ cycles against uncoated Ti-6AI-4V in dry air. X327.

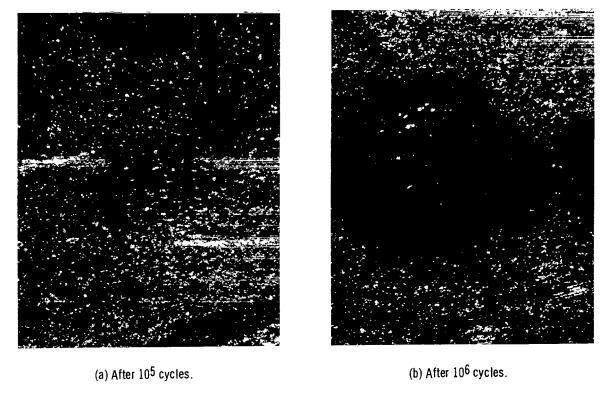


Figure 10. - Methyl phenýl silicone bonded MoS_2 after fretting against uncoated Ti-6Al-4V. X35.

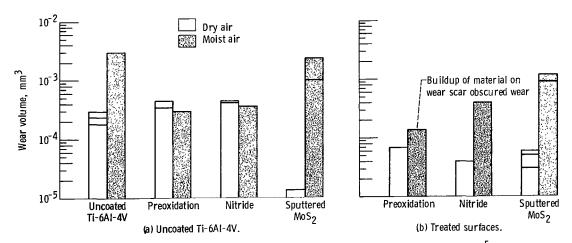


Figure 11. - Fretting wear to untreated Ti-6Al-4V and to surfaces with various treatments after $3x10^5$ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; room temperature.

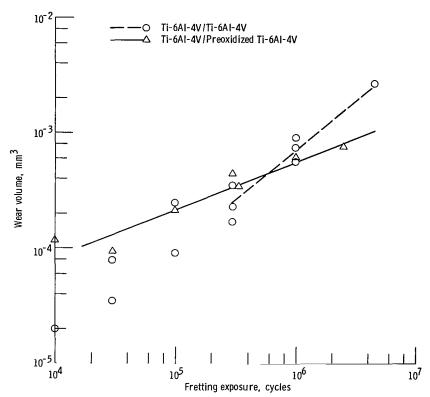


Figure 12. - Fretting wear volume as function of number of cycles for untreated Ti-6AI-4V against preoxidized Ti-6AI-4V. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; dry air; room temperature.

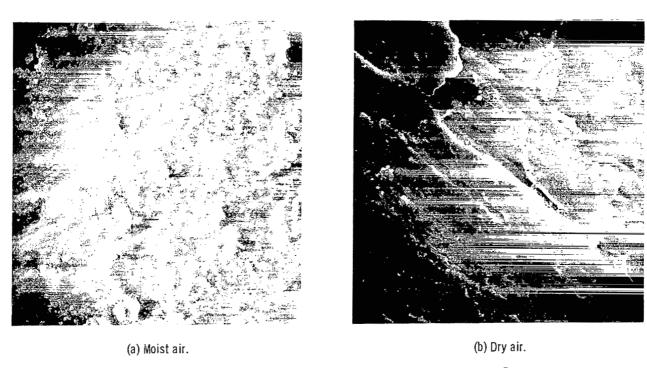


Figure 13. - SEM micrographs of fretting wear scars on sputtered-MoS $_2$ -coated Ti-6AI-4V after $3x10^5$ cycles against uncoated Ti-6AI-4V. x1000.

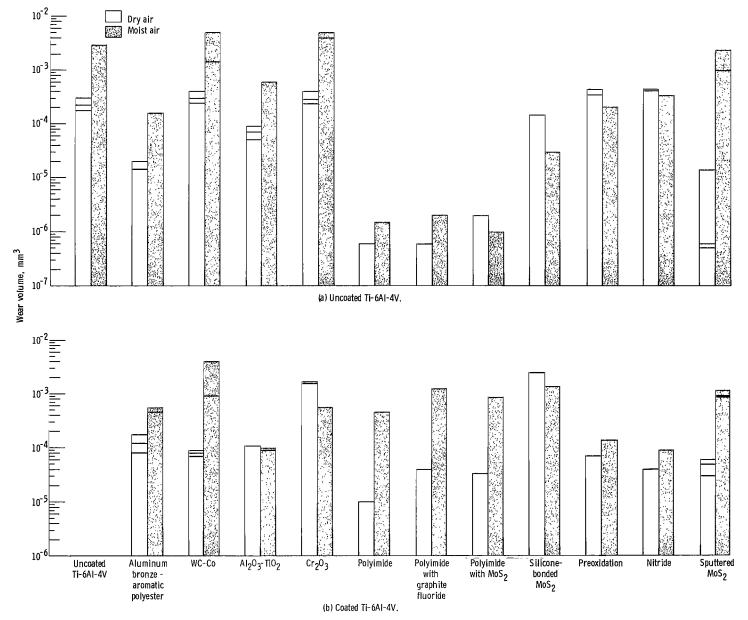


Figure 14. - Fretting wear of uncoated Ti-6Al-4V surfaces and mating surfaces with various coatings and surface treatments after $3x10^5$ cycles. Normal load, 1.47 newtons; frequency, 80 hertz; amplitude, 70 micrometers; room temperature.

OFFICIAL BUSINESS
PENALTY FOR PRIVATE USE \$300

SPECIAL FOURTH-CLASS RATE BOOK



447 001 C1 U C 760409 S00903DS DEPT OF THE AIR FORCE AF WEAPONS LABORATORY ATTN: TECHNICAL LIBRARY (SUL) KIRTLAND AFB NM 87117

POSTMASTER:

If Undeliverable (Section 158 Postal Manual) Do Not Return

"The aeronautical and space activities of the United States shall be conducted so as to contribute ... to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS:

Information receiving limited distribution because of preliminary data, security classification, or other reasons. Also includes conference proceedings with either limited or unlimited distribution.

CONTRACTOR REPORTS: Scientific and technical information generated under a NASA contract or grant and considered an important contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities. Publications include final reports of major projects, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION

PUBLICATIONS: Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION OFFICE

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546