WEAR OF TITANIUM ALLOYS UNDER REPETITIVE IMPULSIVE LOADING

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Abstract

As efforts to conserve energy intensify, so too do research studies in tribology (friction, lubrication and wear). Wear of materials is of basic significance, as components are "lost" in only three basic ways: fracture, obsolescence and wear. Loss of components due to wear is particularly costly; estimates are $100 billion per annum in the U.S. alone. But while a large number of materials have been studied for wear resistance, few include Ti-alloys, yet these are unique and important in a number of applications (e.g., resistance to fretting and erosion). In sliding contact in typical environments, surface layers (e.g., oxides) form; these can break up under increased load. It is clear that mechanochemical reactions determine an equilibrium material "pair" configuration in "dry-friction" sliding impact wear. Accordingly, the structure, morphology and constituents (α,β) of particular Ti-alloys will determine specific wear behaviors. This paper presents results from studies on a variety of Ti-alloys under repetitive impulsive loading. Evaluation by several analyses is presented, including weight loss, light microscopy, SEM and TEM (of worn specimen surfaces and substrates), X-ray diffraction (of wear debris), and EDX (of wear surfaces). In addition, a newly introduced technique for "quantitative" monitoring of material transport is described. Experiments indicate the degree of transverse sliding (which accompanies normal impulsive contact) controls material removal under certain conditions.

Introduction

A large number of machine components is subjected to conditions of relative motion and transmission of loads. Typically, as a result of standard tolerances, an impulsive load accompanies sliding motion (e.g., gears, cams and followers). Such loading is termed compound impact and can lead to wear of opposed surfaces, with resultant loss of service of components and costly "downtime" of machinery.

As the use of titanium alloys in applications of solid contact increases, the need for a clear understanding of tribological behavior becomes necessary. In particular, fretting of titanium alloys as well as severe wear of tools in machining of titanium pose serious technical problems. In general, the friction and wear characteristics of materials are studied by simulating solid contact. In practice, this is accomplished by devices which produce sliding, rolling, or impact (or combinations of these) under various loads and environments. Typically, in a given test environment, the controlling external variables in wear are: (1) relative transverse velocity between mating surfaces, (2) nominal contact stress, and (3) number of cycles or total distance (path length) of frictional contact [1]. The mechanisms by which metals wear are complex (e.g., abrasion, adhesion, fatigue, and corrosion), and are not yet fully
understood. Usually, test conditions themselves influence the role of various basic physical mechanisms. Indeed, for alloys of titanium, no single wear mechanism can be stipulated, although adhesion is usually present.

Also important in the wear of titanium is chemical reactivity. Typically, wear-induced reaction products form and themselves play a role in subsequent mechanochemical processes. Such products may be in the form of oxides, nitrides, or metal alloys, or admixtures which may or may not bond to substrate "base" material.

In early studies under sliding conditions, the effects of varying the alloying elements in titanium were compared and both dry friction and wear behaviors were studied [2,3]. In general, these studies concluded that the alloys of titanium, regardless of alpha-beta phase morphology, exhibited the same poor wear characteristics as did the unalloyed metal. It was further established that hardness, over the range of Rc32 to 41, had no significant influence on wear of the titanium alloy systems tested.

The rationale for the poor wear characteristics of titanium and its alloys was suggested by Miller and Holladay [4]. These authors noted that titanium ordinarily is covered with a layer of "absorbed" gas. This surface condition, termed an "absorbed layer" by these authors, was thought to reduce friction (but not wear) of the metal, and was also noted as being responsible for the nonadherence of ordinary lubricants and most electroplated metallic coatings. In [4] it was noted that material was transferred in either direction between titanium and a host of metallic counterfaces.

The influence of nominal contact stress has been examined at a fixed level of sliding speed [5]. In this study, two distinct modes of wear were found, with a transition at the 3.4 MPa level. The modes were described as oxidative (mild) and metallic (severe), with the oxidative occurring at the lower stress levels.

In wear, relative sliding velocity is manifested at the contact interface in the form of heat. In cases of high speed sliding (e.g., 3 m/s), local asperity temperatures have been estimated to be near 800°C [5,6]. In addition, at a fixed stress level, varying sliding velocity results in distinctly nonmonotonic weight loss behavior. For example, in sliding commercially pure (75A) titanium against 52100 steel in the range 0.5 to 10 m/s, a minimum occurred at 1 m/s and a maximum at 5 m/s [6]. Similarly, in compound impact studies between various Ti (α,β) alloys and 17-4 PH steel, a maximum was found at 2.1 m/s and a minimum at 4.2 m/s (for a fixed stress and number of cycles). In addition, increasing the nominal contact stress caused increased wear of both the Ti alloy specimen and the 17-4 PH steel counterface [7,8].

Clearly, external parameters are significant in creating stress and thermal fields which serve to "process" material in situ in a wear test. By subsequent analysis, it is possible to study the material response to this processing, and to investigate the role of particular wear mechanisms. This paper explores the specific behaviors of various Ti alloys under conditions of repetitive compound impact wear.
WEAR UNDER REPETITIVE IMPULSIVE LOADING

Materials and Methods

As noted in Table 1, a variety of titanium alloys has been studied. This paper focuses particularly on the alloy 5522S (Ti-5Al-5Sn-2Zr-2Mo-0.25Si), with illustrations from other alloys as appropriate. The 5522S is a near alpha alloy with excellent creep strength, thermal stability and high temperature tensile strength. The condition examined is the two phase (alpha-beta) microstructure termed "continuous elongated primary alpha", which is produced by swaging at 870°C followed by air cooling. This microstructure is attractive for wear studies because of the contrasting phase morphology and the potential for surface hardenability through pick-up of Oxygen in the alpha phase.

Table 1

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Type</th>
<th>Composition</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMI-5522S</td>
<td>α,β</td>
<td>Ti-5Al-5Sn-2Zr-2Mo-0.25Si</td>
<td>As received (swaged)</td>
</tr>
<tr>
<td>IMI-685</td>
<td>α,β</td>
<td>Ti-6Al-5Zr-0.5Mo-0.25Si</td>
<td>Solution Treat and Age</td>
</tr>
<tr>
<td>RMI-Beta</td>
<td>β</td>
<td>Ti-45V-1.25Si</td>
<td>Beta Anneal</td>
</tr>
<tr>
<td>Titanium Aluminide</td>
<td>Ti₃Al</td>
<td>Ti-16Al</td>
<td></td>
</tr>
</tbody>
</table>

Titanium alloy specimens are machined to a flat-ended stepped-shaft geometry as depicted in figure 1. Also shown are the relative transverse velocity (V) and normal impacting force (P_max) which act upon the specimen (and counterface). In essence, impact wear testers produce discrete impulsive events which are characterized by the duration (contact time) and maximum force (P_max) as shown in figure 2. The counterface material, in all cases but one, is 17-4 PH martensitic steel. The exception is Ti-5522S, where test alloy specimens were repetitively impacted on the same material counterface.

Prior to testing, both specimen and counterface are polished with SiC paper to achieve a nominal surface roughness of 0.2 µ CLA. Weight (volume) loss data were obtained by weighing specimen and counterface before and after testing to a precision of 0.1 mg.

Results and Discussion

1. Weight Loss

A test series was performed on the RMI 5522S alloy to monitor weight loss for an incubation or run-in period (up to 10,000 cycles) at velocities of 0.01, 0.1, 1 and 10 m/s. At 0.01 and 0.1 m/s the weight (volume) loss following 10,000 compound impact cycles was essentially zero (within accuracy of measurement). Of particular interest are the V=1 and V=10 m/s data which are shown in fig. 3 for both specimen and counterface. Here, the 1 m/s data
17-4 PH STEEL COUNTERFACE MOVES IN X-Z PLANE TO PRODUCE RELATIVE SLIDING AGAINST SPECIMEN

TITANIUM SPECIMEN MOVES IN ± Y DIRECTION TO IMPACT AGAINST COUNTERFACE

Fig. 1 Schematic of specimen and counterface geometry in compound impact wear testing.

Fig. 2 Characteristic impulsive waveform.

Fig. 3 Volume loss vs cycles (distance) for Titanium alloy 5522S specimen and 17-4PH counterface at transverse velocities of 1 and 10 m/s, nominal contact stress = 6.9 MPa.
indicate a higher degree of wear for both specimen and counterface. Interestingly, the 10 m/s data for counterface reveals no weight loss over the first 4000 cycles. This is then followed by increasing counterface weight (volume) loss under continued repetitive load cycling.

2. Microscopy

Following wear experiments, the specimen surfaces are examined by microscopy. In this, the light microscope reveals a unique feature, namely that of brightly colored (blue and straw yellow) oxides. These oxides were observed only on the \(V=10\) m/s specimens and respective counterfaces. The lower velocity specimens appeared metallic or dark grey.

Subsequent to examination by light microscopy, worn specimen surfaces are then prepared for scanning electron microscopy (SEM) and examined from 50 to 30,000 X, with identifying features noted wherever possible. In general, such surface micrographs are similar to those observed with a variety of metallic specimens. Representative examples are shown in figures 4 and 5.

![Fig. 4 SEM micrograph of worn specimen surface at transverse velocity of 10 m/s following 1,000 cycles of compound impact, RMI 5522S.](image)

![Fig. 5 SEM micrograph of worn counterface surface at transverse velocity of 10 m/s following 1,000 cycles of compound impact, 17-4 PH.](image)

Following surface examination, subsurface sections were taken along the sliding direction. Subsequent polishing and etching was employed to reveal microstructural features in specimen and counterface substrates. Representative micrographs are shown in figures 6 and 7 with distinctions noted mainly in the wear surface regions.

An important aspect of subsurface microstructure is the development of wear produced zones. Perhaps the most significant finding [9] is that there are (at most) three characteristic subsurface zones which are depicted
Fig. 6 SEM micrograph of 5522S subsurface region beneath worn surface, $V=10$ m/s, $N=50$ cycles; note early onset of distinct zones.

Fig. 7 SEM micrograph of subsurface region beneath worn friction surface, $V=10$ m/s, $N=100$ cycles, RMI 5522S.

schematically in fig. 8. Zone 1 is the base material, unaffected by wear. Zone 2 is an intermediate region between the contact surface and Zone 1. This is characterized by plastic deformation which ranges from zero at the Zone 1/Zone 2 "interface" to a maximum at the Zone 2/Zone 3 regions. Zone 3 is a thin region with the contact surface as one bound and the Zone 2/Zone 3 "interface" as the other.

Figure 9 illustrates zone formation at the 10 m/s level, together with the increase in microhardness from Zone 1 to Zone 3. At velocity levels below 10 m/s, Zone 3 is less well established, although Zone 2 is observed in all specimens.

3. Plastic Strain Measurement

Zone 2, as previously noted, is a region of plastic strain accumulation. For the 5522S alloy, an assumption of constant volume beta crystals is reasonable. This allows for a measurement of change in length (as referenced to Zone 1 beta crystal dimensions) for the Zone 2 beta crystal population. Results are statistically averaged to yield the data shown in figure 10. The data at $V=1$ m/s indicate plastic strains which range from 75% at a depth of 35 microns to a maximum of 400% at 5 microns from the surface. A still more dramatic effect is observed with the $V=10$ m/s specimen where strains range from 50% at a depth of 12 microns to 600% at 4 microns. Measurement of plastic strain in the Zone 3 region is not possible due to the fracture of the beta crystallites.

4. Zone 3 Characterization

Basically, there are three ways in which to study Zone 3: (1) surface analysis, (2) subsurface analysis, and (3) wear debris analysis. One aspect of surface analysis is the investigation of material transport or adhesive
WEAR UNDER REPETITIVE IMPULSIVE LOADING

Fig. 8 Schematic of subsurface zones which are produced during the wear experiment (in situ) in both specimen and counterface.

Fig. 9 SEM micrograph of V=10 m/s, N=1,000 cycles 5522S specimen subsurface with zones identified; note the increase in microhardness as wear surface is approached from Zone 1 to Zone 3.

Fig. 10 Plastic strain, Zone 2, at V=1 m/s, N=50 cycles, nominal contact stress = 6.9 MPa, also Plastic strain, Zone 2 at V=10 m/s, N=10 cycles, nominal contact stress = 69 MPa.

Energy Dispersive X-ray (EDX) analysis has been employed on worn specimen and counterface surfaces. This allows for the monitoring of material transport phenomena which occur in sliding surface interaction. The technique allows the determination of elemental composition of Zone 3 to a depth of approximately 1000 Å. In order to achieve non-localized results, all EDX data are obtained at low magnification (60X) such that the sampling area is representative of the surface state of Zone 3. Representative data are shown in fig. 11 for the 10 m/s velocity level. This information indicates transfer between tita-
nium and steel during run-in as well as in equilibrium surface states which form as load cycling continues.

![Graph](image)

**Fig. 11** Material transport vs cycles plot at $V=10$ m/s, nominal contact stress $= 6.9$ MPa.

The second form of Zone 3 analysis is subsurface examination. Standard SEM techniques do not allow for resolution of structure in this near-surface layer. An alternative method is the replica technique employed with the higher resolution of the Transmission Electron Microscope (TEM). Figures 12 and 13 show the replicated Zone 3 regions for the titanium alloys RMI 5522S and IMI 685 respectively. In both cases, the transition from Zone 2 to Zone 3 is seen to embody the disintegration and mixing of beta crystals.

The IMI 685 alloy was chosen for thin foil viewing of the near-surface region because of the ease of viewing dislocation structures in the larger grain material. Figures 14 and 15 show the structures of Zone 1 (no wear region) and Zone 3 (worn surface) respectively. The structure near the wear surface (Zone 3) (fig. 15) no longer resembles the unworn (Zone 1) α,β morphology, and further gives evidence of significant subsurface plastic deformation with dense dislocation regions observable throughout the micrograph.

The third form of near surface (Zone 3) investigation is the analysis of wear debris. Clearly, such debris arises from Zone 3, and in some cases, from both Zones 2 and 3, or from all three Zones. In any case, particulate matter may be subjected to subsequent reactions (e.g., oxidation) following initial removal from a substrate. Thus debris properties need not always reflect the material state in Zone 3. Similarly, particles from Zones 2 and 3, primarily consisting of mechanically deformed material from specimen and counterface, may undergo structural transformations due to locally high temperatures at the friction interface.

In practice, debris is collected following wear testing from a smooth paper placed 25 cm beneath the spot of impact. Inspection of particle size and shape is by SEM and X-ray diffraction. Occasionally, debris is separated magnetically.
The particle size of the debris varies with the material pair and with external parameters. Average lengths of 0.3 to 0.7 mm have been observed. In general, the shape of the particles is platelet-like, and X-ray diffraction of some of these reveal preferred crystallographic orientation (texture).

With respect to the amount (ratio) of the various constituents in the debris, it must be noted that the mode of collection allows only a rough estimate. Furthermore, neither traces of crystalline phases nor amorphous constituents can be recorded by the X-ray powder method. Nevertheless, overall observations (Table 2) clearly reveal (1) that debris often stems from both
Table 2 Wear Debris Analysis* After Impact Sliding by X-ray Powder Diagrams (counterface 17-4 PH Steel)

<table>
<thead>
<tr>
<th>Specimen Material</th>
<th>Relative Transverse Sliding Velocity (m/s)</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Fe</th>
<th>a-Ti</th>
<th>a-Ti</th>
<th>a-Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>5522S</td>
<td>0.01</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td>(a,b)</td>
<td>0.1</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td></td>
<td>1.0</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td></td>
<td>2.1</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td></td>
<td>4.2</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td></td>
<td>6.3</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
<tr>
<td></td>
<td>10.0</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Fe</td>
<td>a-Ti</td>
<td>a-Ti</td>
<td>a-Ti</td>
</tr>
</tbody>
</table>

* underlined = major amount
parenthesis = minor amount

With respect to the ratio of the various constituents of the wear debris, the data of Table 2 demonstrate a strong dependence on the transverse velocity of impact sliding. However, without further extraction procedures, the X-ray diffraction analysis does not reveal whether there are coherent debris particles containing material from both specimen and counterface. Moreover, a peculiar problem arises with the oxidation products found in the debris, in that both finely dispersed iron and titanium are highly pyrophoric. Thus, at high velocities, spark formation (flashing) can be observed in wear tests with steel or titanium alloys. In fact, the effect of increasing velocity (increasing energy density at the wear interface) can be seen in the debris analysis in that the amount of $\alpha$-Fe$_2$O$_3$ also increases (Table 2).

The occurrence of $\alpha$-Fe$_2$O$_3$ in dry friction of steels (and cast iron) as one component of the material pair is well known [12]. More recently, it has been shown [13] that at low stress levels, $\alpha$-Fe$_2$O$_3$ can be detected in debris even at very low sliding velocities (0.069 m/s).

At this point, it is useful to briefly consider the oxidation behavior of titanium and titanium alloys outside the context of wear. In an environ-
In wear studies, titanium oxides (oxinitrides) are evident at the worn surfaces from characteristic colors (blue or straw yellow) which indicate the role of high temperature phenomena in the creation of new products formed in situ. While some investigators [15,16] have discussed the fact that oxide films might serve as "solid lubricants", often the reaction products from mechanochemical processes are undetected and their role in wear behavior is consequently ignored. Detection is, of course, difficult because of the relatively minor fraction of reaction product to original metal in particulate debris. In addition each material reacts in a unique way with the experimental environment. For example, alpha titanium alloys easily "pick up" oxygen, since the solubility of oxygen in the alpha titanium lattice is very high. This creates a hardening effect in the near surface microstructure (see figure 9). And while this hardened layer is probably very thin initially, its role in the subsequent attrition of material may be quite considerable. In contrast, beta titanium contains smaller voids in its lattice, and consequently does not accommodate such a large amount of oxygen. One therefore expects less of a hardening effect on the "interface layer" (Zone 3) which forms in situ during a wear experiment.

As can be seen from Table 2, the series of titanium alloys tested against 17-4 PH steel at V=2.1 m/s produces debris particles which contain an oxide of γ-iron (austenite). The largest amount of austenite is obtained from debris arising in tests of Ti3Al against the steel counterface at V=10 m/s. This debris was further analyzed by magnetic separation. By this procedure, it was established that the ratio of α-Fe solid solution and γ-Fe solid solution does not change significantly following separation. Thus, it can be concluded that the two iron phases are intimately associated. Here, it is noteworthy that to transform the martensitic steel to form austenite, a temperature of at least 500°C must be reached during the wear process. In sum, iron is present in debris either as the transformed γ phase or as the α phase, and in certain conditions, as in the α-Fe2O3 oxide.

Conclusion

Various titanium alloys have been tested in air under conditions of impact sliding wear. Mechanochemical reactions between specimen, counterface and environment determine equilibrium material structures (subsurface zones) under specific test conditions of sliding velocity and contact stress.

This investigation has highlighted methods of analysis which identify characteristic behaviors in the wear of titanium alloys. A primary finding is that subsurface microstructural study reveals (at most) three characteristic zones, namely: Zone 1, the (unaffected) substrate; Zone 2, an intermediate region between the surface (Zone 3) and Zone 1, which is characterized by accumulated plastic strain; and Zone 3 which is bounded by the friction surface and Zone 2. Zone 3 is composed of highly distorted crystals in a "mixed state" established from the initial material pair and the environment.
Such zones form quickly [17] and are manifested by a variation in hardness, increasing from the substrate (Zone 1) to the surface (Zone 3).

X-ray diffraction and microscope inspection of debris indicate (1) the debris originates from all three zones and (2) oxidation and phase transformation are present when the sliding velocity is of sufficient magnitude.

References


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