Frictional and Wear Behavior of Commercially Pure Ti, Ti-6Al-7Nb, and SUS316L Stainless Steel in Artificial Saliva at 310 K

Eri Miura-Fujiwara¹,*, Tetsuya Okumura² and Tohru Yamasaki¹

¹Graduate School of Engineering, University of Hyogo, Himeji 671-2280, Japan
²Department of Mechanical Engineering, Nagasaki University, Nagasaki 852-8588, Japan

Frictional and wear behavior of SUS 316L stainless steel, commercially pure Ti (Grade 2), and Ti-6Al-7Nb in artificial saliva and in deionized water were investigated at 310 K using a ball-on-disc type frictional test machine with a Ti counterface. The present study aims to obtain information on the in vitro frictional behaviors of Ti-based dental metallic materials, and to investigate the morphological and compositional changes of the surface damages caused by wear and friction. The surface morphology and chemical composition of the wear track was investigated. The results obtained from the frictional test indicates an interpolated dynamic frictional coefficient ranging between 0.3 to 0.5 in both water and artificial saliva, which suggests that solid contact was predominant. The volume loss in water was always higher than that in artificial saliva and the volume loss of Ti-6Al-7Nb and SUS316L decreased with increase in the applied dead load. Microstructural observations suggest adhesive wear to be dominant in specimens tested in artificial saliva, whereas sharper grooves and cracks were frequently observed in the specimens tested in water. The results indicate that surface embrittlement was caused by oxygen, and the strength of the materials was related to the wear and frictional parameters. [doi:10.2320/matertrans.M2015094]

(Received June 2, 2015; Accepted July 3, 2015; Published August 21, 2015)

Keywords: sliding wear; surface microstructure; biomaterials; commercial purity titanium; titanium-aluminum-niobium alloy; stainless steel

1. Introduction

Ti and Ti-based alloys are desirable as biomaterials on account of their good biocompatibility, excellent corrosion resistance, and low density. Thus, Ti and Ti-based alloys are expected to play an important role as biomedical and dental materials. Nowadays, Ti and Ti-base alloys have become important biomedical materials, especially for patients with metal allergies. In addition, they are being used in a wide variety of medical and dental devices, such as artificial joints, dental crowns, inlays, dentures, and orthodontic devices. Despite the advantages of Ti as a biomaterial, there are challenges in terms of material processing. Some of the problems in using Ti as a biomaterial include its poor wear and frictional properties.

According to Rabinowicz,¹¹ there are four major types of sliding friction, including adhesive wear, abrasive wear, corrosive wear, and surface fatigue wear. Adhesive and abrasive wear occur at the contact surface sliding with or without corrosive atmosphere; however, corrosive wear can occur when sliding wear occurs under wetting, of which is so-called tribocorrosion, and recently become a focus of attention in biomaterials field.²³ In addition, repeated sliding can cause fatigue wear. Since there is body fluid or saliva that can act as a lubricant and an electrolyte under internal or intraoral conditions, it is assumed that very complex phenomena of friction and wear could occur. For example, a tooth undergoes friction because of occlusion and the muscular strength of mastication is reported to be approximately 500–700 N.³ Under such severe conditions, various changes can occur on the surface of the tooth or the dental prosthesis and the material would be worn away during wear.³ In the case of orthodontic devices, the intraoral wear would lead to the modification of the contact area between the arch wire and the bracket.⁴–⁸ The fracture of dental implant fixtures could be attributed to fatigue wear and thus failure due to excessive wear requires replacement of the implant. In addition, the wear debris generated can cause a negative response in the body, such as inflammation or osteolysis, which results in implant loosening.⁹–¹⁵ Surface modification, alloying, and heat treatment are common approaches used to improve the wear resistance.³,⁹,¹⁶–²⁴

In the light of wear and friction phenomena in medical or dental devices made of Ti and Ti-alloys, the inferior wear properties affect the durability and quality in the production process. For example, it is well-known that the grinding efficiency of dental devices made of Ti using carborundum or alumina grinding tools is significantly worse than that of other dental alloys, such as Co-Cr alloy. Further, there is also difficulty in obtaining sufficient mirror finished surfaces with conventional silicone rubber point, because of the low thermal conductivity of Ti and its high reactivity with SiC or Al₂O₃ particles.²⁵ Therefore, although it is generally recognized that wear properties are mainly related to the hardness of the materials, they are also affected by other mechanical, chemical, and physical properties and usage conditions, including the combination of counterfeit materials and lubrication.

Combination of two bodies and type of lubricant determine dental devices’ tribocorrosion behaviors, of course. Souza²⁶ reviewed that researches regarding Ti’s tribocorrosion behaviors in oral environment, and mentioned that the mechanism of Ti device changes depending on the hardness of contact material. A dental implant consists of a couple of parts, such as a root fixture, an internal screw, an abutment, and a crown, and which made from wide variety of combination among various materials.²⁷–²⁹ Generally, commercially pure titanium (CP Ti), Ti-6Al-7Nb, Ti-4Al-6V, or ZrO₂ is used for root fixture, and SUS316L or Co-Cr-Mo alloys also have been used. Ti alloys, such as Ti-4Al-6V are major materials for internal screw. Screw loosening³⁰ and breakage due to fretting fatigue are the major problems since Ti and its alloys

*Corresponding author, E-mail: emiura@eng.u-hyogo.ac.jp
are commonly inferior to wear resistance.27,31,32) Regarding orthodontic dental devices, wear and friction between a bracing and an orthodontic wire is also one of the problems in dental field. Orthodontic wire is made from metal such like Ti-Ni alloy, Co-Cr alloy, β-Ti alloy (TMA; Ti-11Mo-6Zr-4Sn), Ti-6AL-7Nb, stainless steel, and so forth.6,33,34) Bracing bracket consist of stainless steel, polycarbonate, and alumina and so forth.5-8,35) There are literatures investigating wear behaviors of the possible combination consisting a root fixture and a screw, or orthodontic wire and bracing bracket, however, they mostly focused on morphology of worn surface, mechanical parameters such as hardness, and wear properties such as wear rate and frictional coefficient. Since Ti is a highly reactive element once its passivation film is removed, thus effect of absorbing of lubricant’s elements, especially O, also should be taken into account. For orthodontic devices, saliva will play an important role as a lubricant.

Therefore, this study investigated the fundamental sliding wear and frictional behavior in artificial saliva and deionized water, and chemical composition at worn surface of Ti-based dental materials, including commercially pure (CP) Ti and Ti-6Al-7Nb in vitro, and discuss relationships among wear parameters, applied load, and O-induced embrittlement by solid-solution with oversaturated O and/or oxide formation due to wear to understand the wear mechanism. SUS316L was also investigated as a control experiment in terms of possible combination of orthodontic devices, and because transfer of the elements in counterface will be easily detected. The microstructures of the worn surfaces were also observed to obtain the relationship between the wear behavior and surface microstructure.

2. Material and Methods

SUS316L (Nilaco Co.), CP Ti (ASTM Grade II, Selec), and Ti-6Al-7Nb (composition mass\%: Ti : Al : Nb = 86.5 : 6.0 : 7.0, GC) ingots with a diameter of 20 mm (SUS316L) and 30 mm (CP Ti and Ti-6Al-7Nb) were used for this study. As a counterpart a Ti ball with a diameter of 5 mm was used. CP Ti and Ti-6Al-7Nb consisted of an equiaxed α phase and lamellar α + β phases, respectively. The ingots were sliced into circular plates of approximately 1 mm in thickness by using a cutting wheel. The surfaces of the specimens were polished with 0.3 μm alumina powder. The initial surface roughnesses (Ra) of the polished SUS316L, CP Ti, and Ti-6Al-7Nb were 1.088 μm, 0.808 μm, and 0.984 μm, respectively.

The hardness of the polished surface was measured by the Vickers’ hardness test. The test load was 2.94 N and the retention time was 10 s. Pin-on-disc frictional tests were carried out for investigating the tribological properties. The details of the friction test machine are given elsewhere.35) The experiments were performed in artificial saliva (Saliveht®) and in deionized water at 310 K. The chemical composition of Saliveht® is shown in Table 1. In this paper, the artificial saliva samples will be abbreviated as AS. The loads (W) of 0.98–4.90 N were applied via dead weight. The rotation radius (r) and rotation speed (ω) were 1 mm and 95.5 rpm, respectively and thus; the liner velocity was 10.0 mm/s. The running time (t) was 5.40 ks and 21.6 ks. The coefficient of friction (μ) was computed based on the frictional force (F) and W. The mean frictional coefficient (μm) was defined as an extrapolated value from the plot of the stabilized μ against the running distance (L). After the wear tests, the surface roughness values and surface contours were measured by a surface roughness-measuring instrument (Kosaka Laboratory Ltd. SE-30D). The area of the worn surfaces was calculated using the surface profiles.

The wear surfaces were observed using an optical microscope (OM) and a scanning electron microscope equipped in an electron probe microanalyzer (EPMA, JEOL JXA-8621MX). Secondary electron (SE) images, Backscattered electron (BS) images, and combined images between topographical and compositional images (Topo + BSI). The chemical compositions of the worn surfaces were also measured by Auger electron spectroscopy (AES, JEOL JAMP-7100E) and by using the EPMA. In AES, the etched depth is expressed by the number of cycles and one Ar etching cycle lasted for 10 s. The confirmed etching depth was approximately 6.7 nm per a cycle in SiC.

3. Results and Discussion

3.1 Vickers’ hardness of the alloys

Hardness is considered as a factor that determines the wear properties of a material. Thus, the Vickers’ hardness (HV) of the alloys was measured, as shown in Fig. 1. As seen in this figure, Hv of Ti-6Al-7Nb exhibited the highest value and was two times higher than the value shown by CP Ti. Hv of CP Ti was marginally higher than that of a tooth enamel tissue. Both the alloys investigated in this study exhibited higher hardness values than CP Ti. Thus, it can be presumed that the wear resistance of Ti-6Al-7Nb will be the highest among these materials.

3.2 The effect of lubrication and dead load on the frictional coefficient

Figure 2 shows the representative μ-L curves obtained from the pin-on-disc tests under a dead load of 4.90 N. In this figure, similar μ-L curve forms were obtained from each of the materials irrespective of the lubricant, indicating that μ increased rapidly at the initial stages and subsequently, a steady-state region appeared. Therefore, solid contact friction is likely to be dominant.1) The temperature increase and
fracture of the passive films occurred during the initial stages, while fracture and formation of a passive film (or oxidation) competed in the steady-state region. In the steady-state region shown by SUS316L, the lubricant temperature was clearly lower than that in water, although differences in the values caused by the lubricant were not as clear-cut in either CP Ti or Ti-6Al-7Nb. Thus, the results suggest that the lubricating effect of AS is superior to that of water, at least for SUS316L.

The values of $\mu$ in the steady-state region were around 0.45 at 4.90 N; however, the value of $\mu$ at 0.98 N was less than 0.1. Hence, hydrodynamic lubrication occurred at 0.98 N. In addition, an abrupt drop in $\mu$ was observed after $L = 1.0 \times 10^4$ mm within the steady-state region at 2.94 N. The curve at 2.94 N increased up to above $\mu = 0.5$ initially and then dropped down to around $\mu = 0.1$, although the steady-state regions at 0.98 N and 4.90 N seemed to be rather constant. These results indicate that the frictional mode changed from around 2.94 N in AS.

From the $\mu$-$L$ curves, an extrapolated friction coefficient ($\mu_0$) of the alloys was plotted against the dead load $W$. Relationships between $\mu_0$ and the dead load are shown in Fig. 4. In this graph, the values of $\mu_0$ of SUS316L and CP Ti in water, and that of Ti-6Al-7Nb in water and in AS under the entire load ranged between 0.3 and 0.6. This value is sufficiently low for mild wear behavior. According to literature, large irregular fluctuations in $\mu$ plotted against the running distance in the steady-state region as seen in CP Ti in water, Ti-6Al-7Nb in water and in AS in Fig. 2, indicates the dominant severe frictional behavior, although
the obtained friction coefficients were mostly in the range 0.3–0.6, as seen in Fig. 4. This behavior is generally observed when the two sliding surfaces consist of the same metal or compatible materials, as shown by the substantial solubility of one of the metals in small quantities of the other. Besides, the values of $\mu_0$ of SUS316L in water ranged from 0.6 to 0.8 and showed the highest value among all alloys at each dead load. This indicates that AS is more effective as a lubricant than water in the case of SUS316L, whereas the various lubricants did not affect the friction coefficient in the case of Ti-6Al-7Nb.

On the other hand, $\mu_0$ is generally independent of the dead load; however, transition appeared in samples of CP Ti tested in AS at a load of 2.94 N in which the $\mu$-$L$ curve exhibited instability, as mentioned in Fig. 3. In Fig. 4, $\mu_0$ was less than 0.1 at 0.98 N and 2.94 N, indicating that hydrodynamic lubrication occurred below 2.94 N. Therefore, the result indicates that AS is a better lubricant than water in the case of SUS316L, and CP Ti only in the low applied dead load regions. In addition, this abrupt drop of $\mu_0$ may suggest that the threshold load of the oxide layer breakage exists around 2.94 N. It can be assumed that hydrodynamic lubrication occurred on the surface covered with the hard layer such like oxide layer because of the presence of AS since the oxide layer breakage cannot occur under this load.

Even though the load dependence of $\mu_0$ was seemingly unclear except in the case of CP Ti in AS, $\mu_0$ at 2.94 N tended to show a higher value than that at 4.90 N. In other words, $\mu_0$ tended to show the maximum or minimum value at 2.94 N. This phenomenon could have been caused by the experimental conditions. This might have been caused because of the occurrence of severe work hardening due to wear since the rotation radius was smaller than the ball diameter. It can be assumed that the higher the load applied, the more concentrated was the wear on the surface.

### 3.3 Wear properties of the test materials in liquid

Figure 5 shows the variation of the proportional volume loss of the alloys, $V/WL$, against the dead load, $W$. $V$ is the volume loss and $L$ is the running distance. In this graph, $V/WL$ in AS was clearly lower than that in water for each alloy, although $\mu_0$ in water was not necessarily higher than $\mu_0$ in AS. In addition, $V/WL$ decreased with increase in $W$ except in the case of CP Ti. Among the samples, $V/WL$ of SUS316L was the lowest and that of Ti-6Al-7Nb was the highest of all, regardless of the load dependence of $\mu_0$. $V/WL$ of Ti-6Al-7Nb ranged from $10^{-6}$ to $10^{-7}$ mm$^2$/N through the entire load, and $V/WL$ of SUS316L showed the lowest value in both lubricants at each load and the values were around $10^{-7}$ mm$^2$/N, although $\mu_0$ of SUS316L in water was the highest, as shown in Fig. 4. In the case of CP Ti, $V/WL$ in water was almost identical ($\sim 3 \times 10^{-7}$ mm$^2$/N) to that of Ti-6Al-7Nb in AS. At 0.98 N, $V/WL$ of CP Ti in AS was close to the value in water and to the value shown by Ti-6Al-7Nb in AS; however, the value drastically dropped to around $2 \times 10^{-8}$ mm$^2$/N at 2.94 N. The difference in the $V/WL$ values of CP Ti in water and AS was the largest at 2.94 N and 4.90 N, in comparison to other alloys. The volume loss decreased continuously with increase in $W$, with the exception of Ti-6Al-7Nb or CP Ti in AS.

In terms of hardness, $H_v$ of Ti-6Al-7Nb is the highest among the tested alloys, as seen in Fig. 1. However, in terms of the $V/WL$ values, Ti-6Al-7Nb showed the highest and SUS316L showed lowest values. The representative mechanical properties of CP Ti, Ti-6Al-7Nb, and SUS316L are shown in Table 2. As shown in this table, the elongation of SUS316L is the highest and that of Ti-6Al-7Nb is the lowest among the materials tested. The yield stress and hardness of Ti-6Al-7Nb are the highest; however, the elongation or ductility is important in determining the wear. Of course, the results also suggest that the surface embrittlement due to corrosion, oxidation and/or forming solid solution with O during wear should be taken into consideration. Microstructural observations of the worn surfaces will be required to understand the relationship between the wear parameters and wear damage.

### 3.4 Surface morphology

The results of the friction tests revealed that the dead load and the lubricant to some extent affected the friction and wear parameters. However, it is also important to consider if the wear track morphology changed because of the load and/or lubricant. Figure 6 shows the surface morphologies and
surface profiles of the wear tracks obtained in water or AS under a load of 2.9 N. Comparing each surface morphology and wear profile with respect to lubricants, the wear tracks in water seemed dull and the surface roughness seemed lower than that in AS. Comparing the dullness of the wear surface, the wear surface of SUS316L was even sharper than CP Ti or Ti-6Al-7Nb. These characteristics of surface morphology suggest that adhesive wear was dominant in water, especially in the case of Ti alloys. On the contrary, wear track marks in AS were clear and sharp, and the depth of surface profile was larger than that in water with the exception of CP Ti in AS. The results suggest that abrasive wear was dominant in the case of SUS316L and Ti-6Al-7Nb under the conditions employed. With regard to the diameter of the wear track, the values of both SUS316L and Ti-6Al-7Nb in AS were close to the respective values in water, although the diameter of the wear tracks of CP Ti in water was much larger than that in AS irrespective of whether $\mu_0$ was greater than or less than 0.1. In addition, the wear track of CP Ti showed two different types of surface profiles. The roughness was deeper when $\mu_0 > 0.1$ than when $\mu_0 < 0.1$ and the wear track at $\mu_0 < 0.1$ was relatively dull or flat in comparison with other alloys. These results suggest that abrasive-like wear is likely to be included when $\mu_0 < 0.1$, and the wear mode integrated or changed toward higher load conditions at $\mu_0 \sim 0.3$.

3.5 Microstructural observation of the wear track

Microstructural and chemical composition analyses were performed using SEM-EDS and EPMA. The BS images the wear surfaces of CP Ti, Ti-6Al-7Nb, and the Ti counterpart tested in water for 21.6 ks at a dead load of 2.94 N are shown in Figs. 7 and 8, respectively. Results obtained when the materials were tested in AS are also shown in Figs. 9 and 10, respectively. In addition, quantitative analysis of the results of EPMA at each position indicated in these figures is shown in Tables 3 and 4, respectively. The chemical composition was converted from mass% to mol%. The SE image of the wear surface of CP Ti tested in water (Fig. 7) indicates that the wear track consisted of scratches with deep steps and shallow bumpy scratches along the wear direction, while the cracks perpendicular to the wear direction on the tracks were observed as dark areas in the BS image. As seen in Fig. 8, the wear track morphology of the Ti-6Al-7Nb surface tested in deionized water seems to be similar to that of CP Ti. Grooves, clear steps, and shallow bumpy scratches extended along the wear direction and fine cutting flutes were observed around the wear direction. Cracks perpendicular to the wear direction were also observed in the dark area on the groove, where several mol% of O was detected, as shown in Table 3. Since the solubility limit of O in Ti is approximately 30 mol%, the quantitative analysis results indicate the formation of a Ti-O solid solution due to wear.

In the case of the CP Ti wear surface tested in AS (Fig. 9), a dark area containing high O content and elements present in the liquid was observed as shown in Table 4. It should be noted that other elements in AS, such as Na, K, Mg, Cl and P, were hardly detected except P which was detected more than 1 mol% in the worn CP Ti surface. Therefore, O was the most induced element into the worn surface even in AS. From the surface morphology observed in this figure, scratches seemed to be partly inhomogeneous and grooves were rarely observed. Thus, abrasive wear was predominant in AS rather than in water. These results are in good agreement with the OM observations shown in Fig. 6. Li et al. have studied the surface morphology of Ti-6Al-4V worn against a SUS pin in
0.9% NaCl.\textsuperscript{40} The surface morphology they described is similar to what we observe in water. Their XPS analysis of the wear surface indicates that the formation of brittle TiO$_2$ on Ti-6Al-4V might cause embrittlement during wear in NaCl solution, which increased the wear loss of the Ti-6Al-4V samples.\textsuperscript{40}

The morphology of the counterface in AS was also different from that in water in comparison to the worn surface in water and in AS, as seen in Figs. 7–10. In water, the Ti ball showed wearing to some extent, whereas a small amount of wear was observed in AS. Therefore, abrasion volumes of the counterface in AS appeared to be smaller than that tested in water and the volume loss of the specimens tested in AS were smaller than those tested in water. Thus, AS is superior to deionized water in terms of wear resistance, although the lubricating ability of AS is not clearly observed to be exceptional from the frictional coefficients shown in Fig. 4.
Figures 11 and 12 show the BS image and Ti X-ray map of the wear tack on SUS316L tested at 2.94 N in water and AS, respectively. The results of the chemical composition measured by quantitative analysis using EPMA are also shown in Tables 5 and 6. A gray area was observed on the surface, as described in previous BS images of CP Ti and Ti-6Al-7Nb, where a large amount of O was detected than the whiter area. Simultaneously, in Fig. 11, Ti was detected around the cracks, voids, and in grooves, which indicates that Ti was transferred from the counterface to the specimen during the wear test. From the results of EPMA in Table 5, the worn area in gray tested in water contained very high O to the extent as to be able to form titanium oxide and/or iron oxide. Ti transfer was also observed in AS, as seen in Fig. 12, and where P and O with high concentrations were detected. According to chemical composition ratio among Ti, Fe and O, formation of TiO, TiO$_2$ and Fe$_2$O$_3$ was inferred on the worn surface. However, more detailed studies should be carried out to conclude their chemical state, since TiO is non-stoichiometric phase and is much less stable than TiO$_2$. In any event, the chemical composition results and morphology of worn surface damage in the gray area such like the cracks,
voids, and grooves suggested that O-induced embrittlement occurred during friction. It can be understood that the differences observed in the O content on the worn surface caused by various lubricants is probably related to changes in the wear and friction mode.

In order to evaluate the damage depth due to friction, the depth profiles of Ti, O, and P concentration were measured by AES. Figure 13 shows the depth profiles of the maximum AES intensity of Ti, P, and O from the top surface of Ti-6Al-7Nb tested in AS under 2.94 N for 21.6 ks. In this figure, the etched depth was expressed by the number of cycles and one Ar etching cycle lasted for 10 s. The etching depth per a cycle in SiC was about 6.7 nm.

The results of the AES depth profiles indicate that the intensity of P slightly increased up until approximately 20 cycles (134 nm) of etching and then decreased gradually until around 70 cycles (469 nm). The intensity of O was stronger than that of P at the beginning, then it decreased gradually until around 150 cycles (1005 nm) and finally the intensity of O became identical to the background level and the intensity of P, indicating that P might be concentrated within a few hundreds of nanometers of depth like a film on the wear surface. O diffused deeper than P through the wear surface and the intensity of Ti rapidly increased with increase in the etching cycles and hence, the intensity of O seemed to decrease linearly with increase in the depth.

Table 3 Chemical compositions measured by EPMA point analysis on the worn surface of CP Ti and Ti-6Al-7Nb in deionized water. The corresponding images are shown in Fig. 7 and 8.

<table>
<thead>
<tr>
<th></th>
<th>CP Ti</th>
<th></th>
<th>Ti-6Al-7Nb</th>
<th></th>
<th></th>
<th>Ti-ball</th>
<th></th>
<th></th>
<th>Ti-ball</th>
</tr>
</thead>
<tbody>
<tr>
<td>mol%</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>Ti-ball</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>Ti-ball</td>
<td>1</td>
</tr>
<tr>
<td>Ti</td>
<td>93.20</td>
<td>80.94</td>
<td>75.92</td>
<td>70.25</td>
<td>83.40</td>
<td>73.28</td>
<td>73.09</td>
<td>86.56</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>0.02</td>
<td>0.00</td>
<td>0.06</td>
<td>0.04</td>
<td>13.32</td>
<td>18.67</td>
<td>17.41</td>
<td>1.11</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>0.03</td>
<td>0.00</td>
<td>0.02</td>
<td>0.02</td>
<td>3.18</td>
<td>2.14</td>
<td>3.19</td>
<td>0.21</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>0.02</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>0.04</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.03</td>
<td>0.00</td>
<td>0.02</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.03</td>
<td>0.00</td>
<td>0.04</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>Cl</td>
<td>0.03</td>
<td>0.04</td>
<td>0.00</td>
<td>0.00</td>
<td>0.03</td>
<td>0.00</td>
<td>0.04</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>6.74</td>
<td>18.98</td>
<td>23.95</td>
<td>29.64</td>
<td>5.88</td>
<td>6.26</td>
<td>12.07</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>K</td>
<td>0.02</td>
<td>0.01</td>
<td>0.01</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>100.993</td>
<td>100.503</td>
<td>104.472</td>
<td>98.243</td>
<td>109.202</td>
<td>106.750</td>
<td>100.0000</td>
<td>94.176</td>
<td></td>
</tr>
</tbody>
</table>

—: less than resolution or no peak detected
4 significant digits for mol%

Table 4 Chemical compositions measured by EPMA point analysis on the worn surface of CP Ti and Ti-6Al-7Nb in AS. The corresponding images are shown in Fig. 9 and 10.

<table>
<thead>
<tr>
<th></th>
<th>CP Ti</th>
<th></th>
<th>Ti-6Al-7Nb</th>
<th></th>
<th></th>
<th>Ti-ball</th>
<th></th>
<th></th>
<th>Ti-ball</th>
</tr>
</thead>
<tbody>
<tr>
<td>mol%</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>Ti-ball</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>Ti-ball</td>
<td>1</td>
</tr>
<tr>
<td>Ti</td>
<td>92.44</td>
<td>45.50</td>
<td>61.43</td>
<td>93.94</td>
<td>59.92</td>
<td>47.93</td>
<td>50.53</td>
<td>58.19</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>0.02</td>
<td>0.30</td>
<td>0.12</td>
<td>0.04</td>
<td>29.36</td>
<td>12.70</td>
<td>11.94</td>
<td>1.13</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>Cl</td>
<td>0.01</td>
<td>0.02</td>
<td>0.04</td>
<td>0.02</td>
<td>0.00</td>
<td>0.12</td>
<td>0.11</td>
<td>0.11</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>0.05</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>7.51</td>
<td>52.69</td>
<td>38.30</td>
<td>5.96</td>
<td>6.88</td>
<td>37.17</td>
<td>35.36</td>
<td>40.56</td>
<td></td>
</tr>
<tr>
<td>K</td>
<td>0.02</td>
<td>0.09</td>
<td>0.00</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>102.948</td>
<td>89.447</td>
<td>108.091</td>
<td>101.630</td>
<td>63.600</td>
<td>101.393</td>
<td>107.618</td>
<td>66.524</td>
<td></td>
</tr>
</tbody>
</table>

—: less than resolution or no peak detected
4 significant digits for mol%

Fig. 11 SEM images of the worn surfaces of SUS316L tested in deionized water; (a) SE image, (b) Topo + BS image, (c) BS image, and (d) EDS Ti map.
The AES depth profiles suggest that surface embrittlement may occur because of corrosion, oxidation, and/or O solid-solution embrittlement by wear under lubrication. Moreover, it is inferred that the effect of AS on the surface embrittlement is weaker than deionized water because of the existence of a P containing surface film. Therefore, comparatively, in the film formed in AS could be more protective than the corroded film formed in deionized water.

4. Summary and Conclusions

The sliding wear behaviors of SUS316L, CP Ti, and Ti-6Al-7Nb were investigated using a ball-on-disk type friction machine in AS and deionized water at 310 K. Wear surface observations and chemical composition analysis were carried out using SEM-EDX, EPMA, and AES. The results obtained are as follows.

1) Frictional coefficient

1.1) Solid contact mainly occurred during the tests. \( \mu_0 \) was around 0.3–0.6 except for CP Ti at 1–2.94 N.

1.2) A distinct lubrication effect by the AS was observed in SUS316L and CP Ti at 0.98–2.94 N. The difference was not as clear-cut in the case of CP Ti at higher loads (2.94–4.90 N) and in the case of Ti-6Al-7Nb.

1.3) CP Ti showed load dependence of \( \mu_0 \) in AS.

2) Volume loss

2.1) Load dependence of the volume loss did not completely correspond to that of the frictional coefficient.
2.2) Ti-6Al-7Nb showed the largest volume loss among the various materials in each lubricant, which is probably attributed to abrasive-dominant wear. On the contrary, the volume loss of SUS316L was the smallest. The results shown by SUS316L indicate that a detached material originating from abrasion was hardly discharged as an abrasion powder since adhesive wear was predominant.

(3) Surface observations and chemical analysis

3.1) Wear surface profiles tested in AS exhibited abrasive-dominant wear, although the profiles in water showed adhesive-like wear. The profile of CP Ti tested in AS at a load of less than 2.94 N, where $H_0$ dropped down abruptly, exhibited an adhesive-dominant wear surface. On the other hand, microstructure observations of the worn surface by SEM indicate that the adhesive surface was more dominant in AS and an abrasive-like surface was more dominant in water.

3.2) (Ti, O) phase or Fe oxide phase was formed on the surface after the test. P was detected by AES on the wear surface tested in AS. O-induced embrittlement by water, and both corrosion and O-induced embrittlement by AS were inferred.

Acknowledgments

This work was partly supported by a Grant-in-Aid for Scientific Research from the Ministry of Education, Culture, Sports, Science and Technology, Japan (no. 12777190). This work was partly performed under the interuniversity cooperative research program of the Institute for Materials Research, Tohoku University. The authors are grateful to the Laboratory for Advanced Materials of Institute for Materials Research, Tohoku University, especially Mr. Yoshihiro Murakami for assistance. In addition, the authors are grateful to Prof. Tsuyoshi Kawazoe and Emeritus Prof. Akira Ura of Nagasaki University for their helpful advice.

REFERENCES


38) D. Steel: *Datasets of Daido’s Titanium and Titanium Alloy*.