Evaluation of Sliding Wear Resistant Property of C.P. Titanium and SP-700 Titanium Alloy Surface-hardened by Ar–5%CO Gas

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Sliding wear resistant properties of C.P. titanium and SP-700 alloy surface-hardened by Ar–5%CO gas were evaluated using a counterpart material of a bearing steel, JIS SUJ-2 steel and Nishihara type of sliding wear testing machine. In the latter, two disk specimens were rotated at different rotating speeds under a given compressive applied load, yielding a sliding ratio of 20%. Wear tests were repeated intermittently for several times, and a respective test time period in each series of wear tests was primarily varied. The mass loss in both disks was measured after each test. Wear resistance of annealed C.P. titanium without surface hardening was inferior to that of annealed SUJ-2 steel, but surface hardened C.P. titanium resulted in superior wear resistence over quench-tempered SUJ-2 steel with a hardness of 720 in Hv. Observation results of worn surfaces in both disks indicate that preferential wear occurred in the convex region of a furrow-like pattern formed by a lathe machining, resulting in a reduction of surface roughness values with wear progress. When a respective test time period was extended to 21.6 ks, adhesive wear took place between worn surfaces in both specimens, and the mass loss ratio in titanium disk increased at a much higher rate compared with that of a respective test time period of less than 14.4 ks. The steel debris torn off from worn surface of SUJ-2 steel disk was observed to adhere to the worn surface in surface hardened C.P. titanium disk. Wear resistant property of surface hardened SP-700 alloy was also superior to quench-tempered SUJ-2 steel.

KEY WORDS: surface hardening; sliding wear resistance; C.P. titanium; SP-700 alloy; SUJ-2 steel; worn surface; adhesive wear; surface roughness.

1. Introduction

While commercially pure (C.P.) titanium and titanium alloys have many advantageous properties over other metallic materials, they have one weak property of poor wear resistance. The wear resistant property is needed in various application fields of titanium materials, of which examples are sliding or rotating parts used in machinery, automobile or medical implants as well as similar parts used in military and aerospace fields. ¹,² In other applications of titanium alloys, wear resistance of Ti–6%Al–4%V ELI (hereafter, percents in chemical compositions of alloys always denote mass%) in an abrasive mud slurry ³ and frictional wear percentages in chemical compositions of alloys always denote wear resistance of Ti–6%Al–4%V ELI (hereafter, sliding or rotating parts used in machinery, automobile or medical implants as well as similar parts used in military and aerospace fields). ¹,² In other applications of titanium alloys, wear resistance of Ti–6%Al–4%V ELI (hereafter, percents in chemical compositions of alloys always denote mass%) in an abrasive mud slurry³ and frictional wear accompanied with fretting fatigue in SP-700 alloy were investigated. ⁴,⁵ As reported by Dong et al., there are several kinds of poor tribological properties in titanium materials such as severe adhesive wear, high friction coefficients, susceptibility to fretting wear and strong tendency to seize, which appear to be related to the inherent nature of titanium.¹,² Several causes for these poor tribological properties of titanium have been proposed and verified experimentally.¹,²,⁶,⁷ One of these examples is electron configuration, and titanium among various metals possesses the lowest value of d-bond character. ⁶,⁷ This indicates that titanium is more active and possesses greater friction coefficient over other metals and alloys. Buckley et al. investigated the relationship between friction coefficients and d-bond character in various pure metals, and confirmed that the greater the percentage of d-bond character, the lower the friction coefficient.⁶,⁷ The other example is related to crystal structure or slip characteristic.⁷ The axial ratio c/a (1.588) in a hexagonal structure of α titanium is much less than the ideal ratio in closest packing (1.633). This induces prismatic and pyramidal slips, while other hexagonal metals such as zinc, cadmium or hafnium exhibit the basal slip and the lower friction coefficient. Values of c/a in α titanium were increased by addition of alloying elements such as tin or oxygen, which enhances basal slip and reduces the friction coefficient. Buckley and Johnson also investigated the effect of addition of aluminum into α titanium on wear and friction because aluminum increased c/a ratio accompanying a reduction in both lattice parameters of c and a.⁷,¹ They confirmed that aluminum additions from 11 to 21% improved wear, friction and adhesion characteristics of α titanium.

In general, wear resistance in various metals and alloys is
improved by the increase of hardness in base alloys or in the surface layer of metals or alloys. The former is basically performed by combination of alloy designing and heat treatment, but it is not always useful in titanium materials because the attainable maximum hardness in titanium alloys is very limited compared with steels. The latter is achieved by application of various surface engineering technologies, which have been mostly studied in titanium materials. In fact, various surface engineering technologies such as anodizing, shot peening, nickel or hard chromium plating, various diffusion treatments or plasma spray have been investigated for improvement of wear resistance of titanium or its alloys. Among them, surface hardening using oxygen gas, which is a diffusion treatment, has been most widely studied in titanium materials, and the wear resistant property of C.P. titanium or Ti–6%Al–4%V alloys surface-hardened by this method was evaluated. Thermal oxidation for surface hardening adopted in these studies was mostly conducted at 1173 K, which accompanied marked oxidation for surface hardening adopted in these studies.2,14) On the other hand, the authors studied surface hardening by conducting the post heat treatment under vacuum during thermal oxidation was utilized for deeper case hardening of titanium alloys, where the oxide layer formed during thermal oxidation was utilized for deeper hardening by conducting the post heat treatment under vacuum.2,14) On the other hand, the authors studied surface hardening of C.P. titanium and titanium alloys in use of CO2 or CO gas, and Ar–5%CO gas was found to be the best mixture gas over Ar–CO2 gas to achieve the marked surface hardening under the lowest oxidation rate in both C.P. titanium and titanium alloys.15,16) Surface hardening in C.P. titanium in use of Ar–5%CO gas is caused by solid solution hardening due to oxygen and carbon in α titanium. On the other hand, surface hardening in α+β or β titanium alloys was affected by both of the microstructural variation in the subsurface region and hardening in α phase accompanied with enrichment of these interstitials.

Sliding wear behavior in metallic materials is widely varied by sliding conditions as well as counterpart materials adopted in wear tests. A pin-on-disk tribometer and Amsler wear test machine of type A135 with wheel-on-wheel configuration have been mostly used for evaluation of sliding wear resistant property of titanium materials. The objective of this study is to evaluate sliding wear resistant property of C.P. titanium and SP-700 alloy surface-hardened by Ar–5%CO gas using Nishihara type of a sliding wear testing machine. This machine has disk-on-disk configuration, which is similar to Amsler wear test machine. A counterpart material used in this test was a bearing steel of JIS SUJ-2 steel, of which hardness was varied by heat treatments such as annealing or quench-tempering. The wear test was repeated intermittently for several times, and the mass loss of both disks was measured after termination of each test. A respective test time period was mainly varied in three series of wear tests conducted in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks. The wear resistant property of surface hardened SP-700 alloy disk was also evaluated in combination of quench-tempered SUJ-2 steel disk. The surface roughness before and after wear tests was measured, and the worn surface was observed and analyzed using SEM and XRD, respectively.

2. Experimental

A titanium material used for evaluation of sliding wear resistant property was mainly C.P. titanium containing oxygen of 0.15% and iron of 0.07%, and wear resistance of α+β titanium alloy, SP-700 with Ti–4.5%Al–3%V–2%Mo–2%Fe (hereafter, noted as SP-700 alloy) was evaluated under a limited test condition. A bearing alloy steel of SUJ-2 steel was used as a counterpart material in the sliding wear test, of which chemical composition was Fe–1.05%C–0.25%Si–0.30%Mn–1.45%Mn. The sliding wear test was conducted using Nishihara type of a sliding wear test machine, which is very similar to Amsler type of a wear test machine specified in ASTM A135 used by Dong et al.11 Two round-shape disk specimens are set in upper and lower positions in contact with each other in the radius direction of disks. The size of disk specimens is 30 mm in diameter and 8 mm in thickness, and a respective disk specimen has a center hole with a diameter of 15 mm. Each disk specimen is connected with a driving rod through the center hole and rotates at different rotating speeds under given applied load, which yields sliding between both surfaces in disk specimens. Lower and upper disk specimens were C.P. titanium or SP-700 alloy, and SUJ-2 steel, respectively, which were machined from commercial C.P. titanium and SP-700 alloy plates, and SUJ-2 steel bar. Applied compressive load was 50 kgf, and the rotating speeds in lower and upper disks were 800 and 640 rpm, respectively. This yielded a sliding ratio (V_l–V_u)/V_l of 20%, where V_l and V_u are rotating speeds of lower and upper disk specimens, respectively. Wear surfaces of rotating disk specimens were instantly lubricated by machine oil during wear tests.

Disk specimens of C.P. titanium and SP-700 alloy were surface-hardened by heating at 1073 K for 21.6 ks in Ar–5%CO gas atmosphere. Hardness distribution profiles in the subsurface region of both titanium materials are shown in Fig. 1, which were measured in an interval of 10 μm from the surface using a Vickers hardness tester (Akaishi Corp., HM-102) with a load of 0.01 kgf. It is found that the maximum surface hardness values in C.P. titanium and SP-700 alloy are 820 and 600 in Hv and that surface hardening layer depths are 80 μm and 120 μm, respectively. Disk specimens of SUJ-2 steel were subjected to both heat treatments of annealing and quench-tempering, which
resulted in Vickers hardness values of 220 and 720 in Hv, respectively. Wear resistance of annealed C.P. titanium without surface hardening was also evaluated in combination with annealed SUJ-2 steel. After surface hardening or heat treatment, disk specimens were precisely surface-polished to eliminate very thin oxide layer with maintaining dimensional accuracy, followed by ultra-sonic cleaning and rinsing in acetone.

Wear tests in a respective combination of disk specimens were performed intermittently and repeatedly for several times. Wear test conditions conducted in various combinations of C.P. titanium or SP-700 alloy and SUJ-2 steel disks are listed in Table 1, where a respective and total test time periods in each series of wear tests as well as total rotation numbers in titanium disks are shown. The first wear test was conducted in combination of annealed C.P. titanium with hardness of 180 in Hv and annealed SUJ-2 steel with hardness of 220 in Hv, and the test was repeated by 4 times with the total test time period of 13.2 ks. The second test was performed in combination of surface hardened C.P. titanium and annealed SUJ-2 steel, and the test was repeated by 5 times with the total test time period of 108 ks. Three series of the wear tests were conducted in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel, and a respective test time period per cycle was primarily varied in each series of wear tests. That is, a respective time period in three series of tests was basically 7.2 ks, 14.4 ks and 21.6 ks, and repeated test numbers were 4, 7 and 5 times, respectively. Total rotating numbers of titanium disks in each series of tests were 3.2×10^6, 1.44×10^7 and 1.44×10^6. The wear test of surface hardened SP-700 alloy in combination with quench-tempered SUJ-2 steel was performed in a respective test time period of 14.4 ks and repeated test numbers of 7 times.

The mass of both disks was measured after each step of the wear test, and the degree of wear was evaluated by the wear loss ratio of ΔM/M₀, where M₀ is the initial mass of the disk and the mass loss of ΔM is the difference between the initial mass of M₀ and the mass after each wear test. The initial mass of C.P. titanium and SP-700 alloy disks ranged from approximately 18.1 to 18.3 g, and SUJ-2 steel disk was approximately 31.5 g. The mass of the disk was measured in an order of 0.1 mg using an electric balance. The surface roughness of disk specimens before and after wear tests was measured using a profilometer (Tencor Alpha-step 500). The width and speed of measurements were 5000 μm and 200 μm/s, respectively, and both values of the average roughness Ra and the maximum roughness Rmax were obtained. Surface appearance of disk specimens before and after wear tests was observed using a scanning electron microscope (SEM), and worn surface observations were basically conducted on the disk specimen subjected to the final test in each series of wear tests. XRD analysis was conducted to identify the debris on the worn surface.

3. Results

3.1. Sliding Wear Test Results

Figures 2(a) and 2(b) show results of the wear test conducted in combinations of annealed C.P. titanium without surface hardening/annealed SUJ-2 steel disks and surface hardened C.P. titanium/annealed SUJ-2 steel disks, respectively. Horizontal and vertical axes in these figures are rotating numbers of titanium disk and the wear loss ratio of both disks, respectively. The wear test in combination of annealed C.P. titanium without surface hardening and annealed SUJ-2 steel disks was conducted for a relatively few total rotating number as shown in Fig. 2(a). The wear loss ratio of C.P. titanium disk increases at the higher rate with rotation numbers than that of SUJ-2 steel disk, resulting in the higher wear loss ratio over SUJ-2 steel disk. This is caused simply by relatively lower hardness in annealed C.P. titanium disk than that in annealed SUJ-2 steel disk. On the other hand, wear resistance of C.P. titanium subjected to surface hardening is much improved as shown in Fig. 2(b), and the wear loss ratio of surface hardened C.P. titanium becomes much smaller than that of annealed SUJ-2 steel. The linear relationships between rotation numbers and the wear loss ratio in log–log scale plots are observed in both materials. The wear loss ratio in surface hardened C.P. titanium is lower by around 80 times than that of annealed SUJ-2 steel.

Figure 3 shows results obtained by wear tests in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks, where respective time periods in wear tests shown in Figs. 3(a) and 3(b) are 7.2 and 21.6 ks, and 14.4 ks, respectively. For a respective test time period of 7.2 ks, the wear loss ratio of SUJ-2 steel hardened by

### Table 1. Wear test conditions conducted in various combinations of C.P. titanium or SP-700 alloy and SUJ-2 steel disks.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Combination of disk specimens</th>
<th>Test time period (ks)</th>
<th>Total rotation numbers</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>lower disk</td>
<td>upper disk</td>
<td>each test</td>
</tr>
<tr>
<td>1</td>
<td>Annealed C.P. titanium</td>
<td>Annealed SUJ-2 steel</td>
<td>2.4, 2.4, 4.8, 3.6</td>
</tr>
<tr>
<td>2</td>
<td>Surface-hardened C.P. titanium</td>
<td>Annealed SUJ-2 steel</td>
<td>21.6×5</td>
</tr>
<tr>
<td>3</td>
<td>Surface-hardened C.P. titanium</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>2.4, 7.2×3</td>
</tr>
<tr>
<td>4</td>
<td>Surface-hardened C.P. titanium</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>21.6×5</td>
</tr>
<tr>
<td>5</td>
<td>Surface-hardened C.P. titanium</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>14.4×6, 21.6</td>
</tr>
<tr>
<td>6</td>
<td>Surface-hardened SP-700 alloy</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>14.4×6, 21.6</td>
</tr>
</tbody>
</table>
quench-tempering is largely reduced compared with that of annealed SUJ-2 steel, but it is still higher than that of surface hardened C.P. titanium. Wear rates in both disks which are the slope in the relationship between rotation numbers and the wear loss rate reduce compared with those shown in Fig. 2(b). On the other hand, for a respective test time period of 21.6 ks, the wear loss ratio of surface hardened C.P. titanium obtained after the first wear test with rotation numbers of $2.88 \times 10^5$ is much lower compared with the wear loss ratio obtained after the same rotation numbers for a respective test time period of 7.2 ks. However, the wear loss ratio of this disk increases at a very high rate with the increase of rotation numbers. The wear rate in quench-tempered SUJ-2 steel under this wear test condition is almost similar to that for a respective time period of 7.2 ks, although the wear loss ratio in the former is slightly higher than that in the latter.

Because the wear loss ratio or the wear rate of surface hardened C.P. titanium was largely varied by respective test time periods of 7.2 and 21.6 ks in wear tests, an additional wear test with a respective test time period of 14.4 ks was conducted, of which result is shown in Fig. 3(b). The wear loss ratio in both disks increases with the increase of rotation numbers, and surface hardened C.P. titanium shows the higher wear rate compared with that for a respective time period of 7.2 ks. The wear rate in SUJ-2 steel is almost same with that for a respective test time period of 7.2 or 21.6 ks, although the wear loss ratio of this disk is higher than those for these test time periods. Consequently, surface hardened C.P. titanium resulted in superior wear resistant property over that of quench-tempered SUJ-2 steel under all wear test conditions, but the relatively high wear rate in this disk was observed for the longest respective test time period of 21.6 ks.

Figure 4 shows the wear test result obtained in combination of surface hardened SP-700 alloy and quench-tempered SUJ-2 steel disks. The former shows the lower wear loss ratio than that of the latter, although difference in wear rates between both disks is small. Values of the wear loss ratio in both disks are lower by over one order compared with those obtained in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks shown in Fig. 3(b). As shown in Fig. 1, the maximum surface hardness in surface hardened SP-700 alloy is around 600 in Hv, which is lower than that of quench-tempered SUJ-2 steel. Nevertheless, wear resistance in the former is superior to that of the latter, which is discussed later.
3.2. Observations of the Worn Surface and Variations in Surface Roughness with the Wear Progress

The surface appearance and surface roughness of disk specimens before and after wear tests were investigated using SEM and a profilometer, respectively. The latter results are summarized in Table 2, where $R_a$ and $R_{max}$ are the average and the maximum surface roughness values, respectively. Figures 5(a) to 5(d) show surface appearances of both disks tested in combination of surface hardened C.P. titanium and annealed SUJ-2 steel disks, where (a) and (b) are before wear tests, and (c) and (d) are after the wear test in total rotation numbers of $1.44 \times 10^6$ under a respective testing time period of 21.6 ks. The furrow-like pattern consisting of numerous parallel and straight lines with a constant space is observed on the surface of non-tested fresh disks of both materials, which appears to be formed during machining of disk specimens because the space between parallel lines corresponds to a pitch of revolution during a lathe machining. Disk specimens rotated to the parallel direction to straight lines observed in these micrographs. Worn surfaces in both disks shown in Figs. 5(c) and 5(d) reveal smoother appearance with a reduction of sharpness in straight lines, indicating occurrence of preferential wear of the convex portion in the furrow-like pattern surface. As seen in No. 1 of Table 2, surface roughness values of $R_a$ and $R_{max}$ in the fresh disk of annealed SUJ-2 steel disk are larger than those in surface hardened C.P. titanium disk, and both values in this disk after the wear test are markedly reduced. On the other hand, surface hardened C.P. titanium shows a very little variation in surface roughness values before and after the wear test.

Surface appearances of both disks tested in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel are shown in Figs. 6(a) to 6(f), where (a) and (b) are before wear tests, and (c) to (f) are after wear tests. Wear test conditions in (c) and (d) are a respective testing time period of 7.2 ks and total rotation numbers of $3.2 \times 10^5$, and those in (e) and (f) are 21.6 ks and $1.44 \times 10^6$, respectively. Surface roughness values of both disks before and after wear tests are listed in No. 2 of Table 2. Surface roughness of steel disk before the wear test is larger than that of surface hardened C.P. titanium disk. Surface appearance of quench-tempered SUJ-2 steel disk before the wear test shown in Fig. 6(b) is very similar to that of annealed steel disk shown in Fig. 5(b). As shown in Figs. 6(c) and 6(d), numerous straight lines observed before the wear test

<table>
<thead>
<tr>
<th>No.</th>
<th>Combination of disk specimens</th>
<th>Before test</th>
<th></th>
<th>After test : total rotation numbers</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$R_a$ ($\mu m$)</td>
<td>$R_{max}$ ($\mu m$)</td>
<td>$R_a$ ($\mu m$)</td>
<td>$R_{max}$ ($\mu m$)</td>
</tr>
<tr>
<td>1</td>
<td>lower disk</td>
<td>Surface hardened C.P. titanium</td>
<td>1.1</td>
<td>7.9</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td>upper disk</td>
<td>Annealed SUJ-2 steel</td>
<td>1.5</td>
<td>11.4</td>
<td>0.6</td>
</tr>
<tr>
<td>2</td>
<td>lower disk</td>
<td>Surface hardened C.P. titanium</td>
<td>0.9</td>
<td>4.3</td>
<td>1.2</td>
</tr>
<tr>
<td></td>
<td>upper disk</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>1.7</td>
<td>10.0</td>
<td>0.3</td>
</tr>
<tr>
<td>3</td>
<td>lower disk</td>
<td>Surface hardened SP-700 alloy</td>
<td>0.9</td>
<td>4.0</td>
<td>1.6</td>
</tr>
<tr>
<td></td>
<td>upper disk</td>
<td>Quench-tempered SUJ-2 steel</td>
<td>0.7</td>
<td>3.1</td>
<td>0.7</td>
</tr>
</tbody>
</table>

Fig. 4. Variations of the wear loss ratio of both disks with rotation numbers in combination of surface hardened SP-700 alloy and quench-tempered SUJ-2 steel disks. A respective test period in the wear test is 14.4 ks.

Fig. 5. Surface appearances of both disks tested in combination of surface hardened C.P. titanium in (a) and (c) and annealed SUJ-2 steel (b) and (d), where (a) and (b) are before the wear test, and (c) and (d) are after the wear test in total rotation numbers of $1.44 \times 10^6$. 

Table 2. Surface roughness values in disk specimens before and after wear tests.
in both disks are worn away and mostly disappears after the wear test. Surface roughness values of $R_a$ and $R_{\text{max}}$ in quench-tempered steel disk markedly reduce after the wear test under this condition, approaching to the same values with those of titanium disk. A larger wear loss ratio in this disk over surface hardened C.P. titanium disk was observed as shown in Fig. 3(a), and this mass loss appears to be caused by wearing off from the convex portion of the steel disk surface, resulting in a reduction of surface roughness values. However, surface roughness values obtained after the wear test in surface hardened C.P. titanium change very little, nevertheless straight lines on the worn surface of this disk are not observed. Surface roughness values in this disk was the lowest among many disks used for wear tests, which may be nearing the resolution limit of SEM, but the true cause for this could not be made clear.

Figures 6(e) and 6(f) show worn surfaces of both disks after the wear test with total rotation numbers of $1.44 \times 10^6$ under a respective testing time period of 21.6 ks. A number of wear debris with a white color is adhered along straight lines on the surface of C.P. titanium disk, while black spots or area are observed on the surface of SUJ-2 steel disk. The regions marked by the circle in Figs. 6(e) and 6(f) were observed in the higher magnification by SEM as shown in Figs. 7(a) and 7(b), respectively. Numerous adherent debris are observed in the area with a white color on the worn surface of surface hardened C.P. titanium disk together with small black spots, and a number of area with large black spots is seen on the worn surface of SUJ-2 steel disk.

XRD analysis was conducted on worn surfaces of quench-tempered SUJ-2 steel and surface hardened C.P. titanium disks. Results are shown in Fig. 8, where (a) is the result of surface hardened C.P. titanium disk after the wear test with total rotation numbers of $3.2 \times 10^5$, and (b) and (c) are C.P. titanium and SUJ-2 steel disks after the test with total rotation numbers of $1.44 \times 10^6$, respectively. Any other metal besides the base metals is not detected in worn surfaces of C.P. titanium disk tested in total rotation numbers of $3.2 \times 10^5$ and SUJ-2 steel disk tested in total rotation numbers of $1.44 \times 10^6$. On the other hand, Fe is detected in the worn surface of C.P. titanium tested in total rotation numbers of $1.44 \times 10^6$. The small black spots and the area with large black spots observed in Fig. 7 appear to be voids. Consequently, steel debris which is worn off from the surface of SUJ-2 steel disk appears to adhere to the worn surface of C.P. titanium. As seen in Table 2, surface roughness values of $R_a$ and $R_{\text{max}}$ in C.P. titanium tested under this condition increase compared with values before the wear test.
while surface roughness values of SUJ-2 steel decrease. The feature of the worn surface appearance and a variation in surface roughness values of surface hardened C.P. titanium disk appear to be associated with a rapid increase in the wear loss ratio observed under this wear test condition, because such worn surface appearances as shown in Figs. 6(e) and 6(f) were not observed in both disks tested under respective test time periods of 7.2 ks and 14.4 ks.

Worn surface appearances observed in both disks tested in a respective test time period of 7.2 or 21.6 ks showed no marked difference between the center region and both edges in the disk surfaces. However, worn surface appearances obtained in the wear test condition with a respective test time period of 14.4 ks were different between both regions in disk surfaces. **Figures 9** (a) to (d), and Figs. 9(e) and 9(f) show worn surface appearances of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks tested in rotation numbers of $1.44 \times 10^6$ under a respective testing time of 14.4 ks, respectively. Parallel straight lines remain in the center region in both disks as shown in Figs. 9(b) and 9(e). On the other hand, the worn surface in both edge regions of C.P. titanium disk consists of two layers with different appearance as shown in Figs. 9(a) and 9(c), and the width of these edge regions is several hundreds μm; one layer formed in the most near to the edge has very smooth and flat surface appearance, and the other layer formed beneath this layer has very rough surface appearance. The width of the former is much narrow compared with that of the latter. The region denoted by the circle in Fig. 9(a) was observed by SEM with the higher magnification, which is shown in Fig. 9(d). The worn surface in the region with rough appearance shows the deformed appearance after occurrence of failure. XRD analysis in this region did not show any other metal except α titanium. Thus, very rough surface appearance observed in both disk edge regions appears to be caused by local failure and heavy deformation, but not by adhesive wear. Figures 9(e) and 9(f) show worn surfaces in the center and edge regions of SUJ-2 steel disk, respectively. The worn surface appearance on the other side of the edge was very similar to SEM micrograph of (f). Parallel straight lines remain in the center region, and very flat and smooth worn surface is observed in the edge region of the disk.

Worn surface appearances of surface hardened SP-700 alloy and quench-tempered SUJ-2 steel disks tested in rotation numbers of $1.44 \times 10^6$ under a respective testing time of 14.4 ks were different between the center and both edge regions similarly to the result shown in Fig. 9. Parallel straight lines remained in the center region of both disks. However, such a worn surface with rough appearance as seen in Figs. 9(a) and 9(c) was not observed in the edge regions of both disks, where the region with very flat and smooth appearance was widely spread. Surface roughness values of both disks are listed in No. 3 of Table 2. Surface roughness values of a respective disk before the wear test are lower than those of a respective disk used in wear tests in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks, and these values after the wear test increase a little. As noted above, the lowest wear loss ratio in both disks was obtained in this combination of disk specimens, and this may result in a relatively small variation of surface roughness values in both disks.

4. Discussion

In this study, sliding wear resistance of C.P. titanium was found to be improved by surface hardening using Ar–5%CO gas, which was superior to that of quench-tempered SUJ-2 steel with a hardness of 720 in Hv. This appears to be primarily brought about by higher surface

![Fig. 9. SEM micrographs of worn surfaces in surface hardened C.P. titanium disk in (a) to (d) and quench-tempered SUJ-2 steel disk in (e) and (f) after the wear test in a respective test time period of 14.4 ks and total rotation numbers of $1.44 \times 10^6$. (a) and (c) are both edge regions and (b) is the center region in titanium disk surface. (d) is the high magnification observation in the circled location in (a). (e) and (f) are the center and the edge location in steel disk surface, respectively.](image-url)
hardness in the former over the latter. Surface hardening in C.P. titanium shown in Fig. 1 is caused by solid solution hardening mostly due to oxygen. Carbon is also enriched in α phase, but its contribution to surface hardening is relatively small because of much lower solubility of this interstitial compared with that of oxygen.\textsuperscript{15,16} The increase of oxygen concentration in the surface layer of C.P. titanium increases $c/a$ value in α titanium, which improves the wear resistance of α titanium through a reduction in friction coefficient and enhancement of the basal slip as noted in Introduction.\textsuperscript{7} The effects of lattice constant or $d$-electron character of α titanium on friction and wear were studied using specimens with extremely smooth surface or very low surface roughness values. The disk specimens used in this study were machined so as to yield the similar level of surface roughness to that of the most commercially used machined parts, and so this level of surface roughness is much rougher compared with that of materials used in basic studies of friction or wear. In such a surface roughness level of the disk specimen, the effect of a lattice constant variation on wear appears to yield a minor contribution to the reduced friction coefficient to improvement of wear resistance, and hardness difference between both disk specimens primarily control the wear behavior.

Wear resistance was evaluated based on the wear loss ratio due to the mass loss in this study, but in practical applications of titanium or steel to various parts or components subjected to wear, the wear loss in thickness in these parts often becomes more important rather than the mass loss. In addition, because of density difference between titanium and steel, an evaluation of wear based on the thickness loss possibly gives rise to different results from that of the mass loss, particularly in a relative evaluation of the wear resistance in both disks. Therefore, the mass loss ($\Delta M$) of a respective disk obtained at each step of the wear tests was converted to the wear loss in thickness ($\delta$) by calculation using the following equation.

$$\delta = \frac{\Delta M}{(2\pi r^2 b \cdot \rho)}$$

where $\rho$ is density of the disk material, and $r$ and $b$ are the radius and the width of the disk specimen, respectively. Figures 10(a) and 10(b) show variations of the wear loss in thickness with rotation numbers, corresponding to results of the mass loss ratio shown in Figs. 3(a) and 3(b), respectively. It is found from comparison of Figs. 3 and 10, where the wear loss difference in thickness between C.P. titanium and SUJ-2 steel disks becomes smaller by the amount of density difference between both disk materials compared with the mass loss ratio difference between both disks. However, the relative relationship of both disks in a wear loss variation with rotation numbers is not varied, keeping the lower wear loss in surface hardened C.P. titanium compared with that of quench-tempered SUJ-2 steel. Values of the wear loss in thickness in both disks shown in Figs. 10(a) and 10(b) are varied in the range from 0.03 to 1.7 $\mu$m and from 3.6 to 52 $\mu$m, respectively, which are almost the same ranges of order with surface roughness values in both disks. In fact, wear in both disks took place in the convex region of wear surface, yielding a reduction of surface roughness values as seen in Figs. 6(c) and 6(d). The maximum surface hardness value of surface hardened C.P. titanium obtained from the surface hardness distribution profile which was measured with a pitch of 10 $\mu$m from the surface is around 820 in Hv, but hardness in the in the depth of a submicron or micron meter order from the surface in this disk is estimated to be much harder over this value because of very steep hardness distribution in the subsurface region. In addition to this, interstitials may diffuse to two directions in the convex region of the disk surface with the furrow-like pattern, which appears to enhance hardening in this region. Consequently, hardness in the subsurface region of surface hardened C.P. titanium disk where wear actually took place appears to be much higher than the maximum surface hardness obtained from hardness distribution profile of this disk, resulting in superior wear resistance in surface hardened C.P. titanium over quench-tempered SUJ-2 steel.

The important result obtained in this study was that adhesive wear took place under the wear test condition with a longer respective test time period of 21.4 ks. The wear loss ratio in surface hardened C.P. titanium disk increased at much higher rate than the wear rate observed under the wear test condition with a respective test time period of 7.2 or 14.4 ks, indicating that adhesive wear may be caused by an extension of a respective test time period in the wear test. It is important to note here that the wear loss ratio of surface hardened C.P. titanium disk obtained after the first
test in the wear test condition with a respective test time period of 21.4 ks is lower by one order compared with that value after the first test in a respective test time period of 7.2 or 14.4 ks. The worn surface appearance observed after the first test in this test condition evidently showed that adhesion already took place at this stage. That is, the steel debris adhered to the worn surface of titanium disk tends to increases the mass of titanium disk, while the wear progress reduces the mass. The resulting mass loss of this disk after the first test may become lower than the mass loss in the case without adhered steel debris, yielding a relatively low wear loss ratio after the first wear test. The increase of wear loss ratio at the higher rate observed in the second or successive wear tests may be caused due to two different mechanisms. The first one is the accelerated wear progress in surface hardened C.P titanium disk due to the existence of steel debris between two rotating disk surfaces, and the second one is the additional reduction in the mass of C.P. titanium disk due to breakaway of some amounts of steel debris adhered on the worn surface besides the mass loss due to wear. Although steel debris was formed in this wear test condition, the value of the wear loss ratio in C.P. titanium disk in this test condition was not higher than those values obtained in other two wear test conditions with the shorter respective test time periods, and the wear loss ratio or the wear rate in SUJ-2 steel disk in this test condition was not higher than those values obtained in other two test conditions. These results appear to make difficult to find a true cause for the increased wear rate in C.P. titanium disk observed in this test condition. To find a true cause for the higher wear rate in C.P. titanium disk obtained in this test condition, it is needed to investigate a variation of the amount of steel debris on the worn surface of this disk in each step of the wear test.

The cause for occurrence of adhesion seems to be associated with an extension of a respective test time period in the wear test, which tends to yield an elevation of the temperature in the subsurface region of both disks through heat accumulation, although this temperature could not be measured. The occurrence of wear always accompanied with plastic deformation, which induces adiabatic heating in the subsurface region of the worn surface for high rotation speed of disks. However, the actual temperature rise in both disks is very limited because of the cooling effect due to continuous supply of lubrication oil on the disk surfaces during the wear test. The debris observed on the worn surface of titanium disk was pure iron, but not Fe–Ti alloy or their compounds, which indicates that the temperature rise near the disk surface was not so high as to cause the chemical reaction between C.P. titanium and SUJ-2 steel. Nevertheless, some amount of the temperature rise was caused through heat accumulation in the whole disk. In fact, warming of disks was felt by hands during handling both disks immediately after each test, and this warming was increased with an extension of a respective test time period. The actual temperature in the convex region of the steel disk surface may possibly rise up over the tempering temperature of SUJ-2 steel which was 453 K, reducing the strength or hardness in this portion of steel disk. This may cause failure or fragment in the convex region, and steel debris may transfer to the worn surface of titanium disk. The straight and parallel row formation of the steel debris on worn surface of titanium disk may be yielded by this process. As reported by Dong et al., a large difference of hardness between both disks may cause the abrasive wear, which is the removal of a soft material from the surface by a hard material moving along the surface under the applied load.

The other possible cause for the temperature rise in the disks may be the temperature increase of the lubricant oil. The present wear test machine enables to supply the falling drop of oil at a constant rate, and the oil is circulating through the oil reservoir during the wear test. The volume of oil reservoir is 4 L which is a large enough amount to prevent the increase of the oil temperature even in the wear test condition with a very long test time period. Consequently, there is very few possibility of the temperature rise or unstable supply of lubricant oil during the present wear test.

Under the wear test condition with a respective test time of 14.4 ks, the peculiar layer consisting of two regions with very flat and rough appearances was observed in both edge regions in the worn surface of surface hardened titanium disk. These worn surface appearances were not observed in both edge regions of SUJ-2 steel disk. It appears that the worn surface with rough appearance observed in surface hardened titanium disk is not caused by adhesive wear because of no detection of the steel debris and also different worn surface appearance from that of adhesive wear. As noted, surface hardening in the edge region of C.P. titanium or SP-700 alloy disk may be much enhanced compared with the center region of the disk, and in particular hardness in the convex region of the furrow-like pattern in the edge region of the disk may tend to become extremely high, resulting in the increase of brittleness in the convex region of the disk edge. As a result, this region may be crushed in a brittle manner, yielding the worn surface appearance as shown in Fig. 9(d). The titanium debris formed by crushing may flow away with lubrication oil. The very flat and smooth worn surface appearance was observed in the most outer region of surface hardened titanium disk, although the width of this region was very narrow. Not only the hardness value but also the applied stress condition and the flow condition of lubrication oil in the most outer edge region of the disk are markedly different from those in other locations of the disk, which may possibly result in this worn surface appearance.

The surface roughness of test materials markedly affects friction coefficient in the wear test. Shi et al. studied friction coefficient in the sliding wear test conducted in combination of thermal oxidation-treated Ti–6Al–4V alloy and UHMWPE (ultra-high molecular weight polyethylene), reporting that it increased with the increase in the surface roughness of tested materials. Both materials disks used in this study were machined in such a surface-finish condition as to be adopted in manufacturing of the most machining parts, resulting in surface roughness values as shown in Table 2. As the increase of friction coefficient enhances wear, the decrease of surface roughness values or a reduction of the height in the convex region of the surface in surface hardened C.P. titanium disk is possibly effective to prevent occurrence of such adhesive wear as shown in Figs. 6(e) and 6(f). The improvement of surface roughness may also reduce true stress actually applied in the convex region.
of the surface, and this also contributes to reduce adhesive wear.

It is found from comparison of Fig. 3(b) and Fig. 4 that values of the wear loss ratio obtained in both disks of surface hardened SP-700 alloy and quench-tempered SUJ-2 steel were much lower compared with those obtained in combination of surface hardened C.P. titanium and quench-tempered SUJ-2 steel disks under the same wear test condition. As shown in Fig. 1, the maximum surface hardness obtained from hardness distribution profile of SP-700 alloy is lower than that of surface hardened C.P. titanium, and so, this result indicates that the wear loss ratio is not controlled simply by hardness, but it is also affected by combination of the test material and a counterpart material. The wear loss ratio of SP-700 alloy is lower than that of SUJ-2 steel although the maximum surface hardness in the former is lower than hardness of quench-tempered SUJ-2 steel. The mass loss in both disks shown in Fig. 4 was converted to the wear loss in thickness similarly to Fig. 10, which is shown in Fig. 11. It is found that difference in the wear loss between two disks becomes not so significant, but that surface hardened SP-700 alloy disk still shows the lower thickness loss than that of SUJ-2 steel. The wear in surface hardened SP-700 alloy disk is varied in the thickness loss under 0.3 μm. As already noted, hardness is such a depth from the surface may be much higher than 600 in Hv which was obtained from hardness distribution profile in this alloy, probably exceeding hardness of quench-tempered SUJ-2 steel. That is, the higher surface hardness in surface hardened SP-700 alloy results in the lower wear loss in the mass or thickness than that of SUJ-2 steel.

5. Conclusions

The sliding wear resistant property of surface hardened C.P. titanium and α+β titanium alloy of SP-700 was evaluated using Nishihara type of a sliding wear test machine and a counterpart disk material of a bearing steel, JIS SUJ-2 steel. Disk specimens of titanium materials were surface hardened by heating at 1073 K in Ar–5%CO gas atmosphere. Wear tests with a sliding ratio of 20% between two in rotating disks were conducted intermittently and repeatedly under oil lubrication, and wear was evaluated by the mass loss ratio. The following results were obtained.

(1) Wear resistance of annealed C.P. titanium is inferior to that of annealed SUJ-2 steel, but wear resistance of surface hardened C.P. titanium is superior over that of annealed SUJ-2 steel.

(2) When a respective test time period in wear tests is 7.2 ks or 14.4 ks, wear resistant property of surface hardened C.P. titanium is also superior to that of water-quenched SUJ-2 steel with a very high hardness of 720 in Hv.

(3) The worn surface of SUJ-2 steel disk shows occurrence of preferential wear in the convex region in a furrow-like pattern formed by a lathe machining, and therefore, surface roughness values of this disk tends to reduce with wear progress.

(4) When a respective test time period is extended to 21.6 ks, adhesive wear takes place between surface hardened C.P. titanium and quench-tempered SUJ-2 steel, which increases the wear loss ratio in titanium disk at a higher rate with the increase of rotation numbers. Steel debris torn off from worn surface of SUJ-2 steel disk was observed to adhere on the worn surface in surface hardened C.P. titanium disk.

(5) Wear resistant property of surface hardened SP-700 alloy is also superior to quench-tempered SUJ-2 steel.

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