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Investigation on interfacial adhesion of Ti–6Al–4V/nitride coatings

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Both 10-meter ball-on-disk tests and 0.01-meter single pass sliding tests were performed under the same test conditions to compare the adhesion mechanism of titanium alloy Ti–6Al–4V to AISI 52100 steel and two nitride coatings deposited by physical vapor deposition technique (PVD), namely, AlTiN and CrN. The sliding surfaces and the interface region between the transfer layer and the counterface surfaces were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), electron energy loss spectroscopy (EELS), and energy dispersive spectroscopy (EDS). The results revealed that the adhesion of titanium alloy to the counterface materials initiated during the initial stage of sliding in unlubricated sliding contact and resulted in the transfer of titanium alloy to the counterface surfaces. It was found that the transferred Ti–6Al–4V had a layered amorphous structure in which nanocrystalline and polycrystalline oxides were embedded. Severe oxidation of titanium was observed in all the tests. However, Ti–6Al–4V showed less severe oxidation behavior when sliding against CrN. High resolution TEM (HRTEM), electron diffraction, and fast Fourier transform (FFT) investigations carried out at the interface region between the transfer layer and the counterface surfaces revealed the presence of nanocrystalline TiO (C2/m) at the transfer layer interface with steel and AlTiN. However only α -titanium (P6₃/mmc) was detected at the CrN/transfer layer interface.

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1. Introduction

Titanium alloys possess remarkable properties, including high specific strength and corrosion resistance, biocompatibility and fatigue strength and have found a wide range of applications in the aerospace, transportation, biomedical, chemical, and petroleum industries. However, their poor wear resistance and susceptibility to galling results in components' failure when sliding contact is unavoidable and thus prevents extensive application of titanium alloys [\[1,2\]](#page--1-0).

Two factors that contribute to the high wear rate of titanium alloys, according to Molinari et al. and Liu et al. [\[3,4\],](#page--1-0) are low work hardening, which results in adhesion, abrasion, and delamination, and the easily-removed oxides formed during sliding, which cannot protect the alloy effectively. They found that the main wear mechanisms for titanium alloys were oxidation-dominated wear and delaminationdominated wear and a minimum wear rate was achieved at medium sliding speeds corresponding to a transition from oxidation to delamination wear.

The effect of the counterface material on the adhesion and wear behavior of titanium alloys has been studied by several researchers. Straffelini and Molinari [\[5\]](#page--1-0) reported that the wear rate of Ti–6Al–4V was higher when sliding against AISI M2 steel, compared to self-mating sliding conditions due to the abrasive action of hard carbide

particles in the steel microstructure. They highlighted that severe oxidation and transfer of Ti–6Al–4V occurred during unlubricated sliding wear of the alloy irrespective of the counterface material and found that the transfer layers and the generated wear debris particles mainly consisted of TiO and α -Ti according to X-ray diffraction (XRD) analyses. A tribochemical wear mechanism was proposed by Dong and Bell [\[6\]](#page--1-0) for Ti–6Al–4V surfaces sliding against alumina. They found that the tribolayers on the surface of alumina counterpart consisted of intermetallics such as $Ti₃Al$, a reduction product of alumina by titanium. They also reported a strong bonding between the tribolayer and the counterface possibly due to the mutual diffusion and formation of the titanium aluminides at the interface.

Qu et al. [\[7\]](#page--1-0) studied the tribological behavior of titanium alloys (Ti–6Al–4V, Ti–6Al–2Sn–4Zr–2Mo) against ceramic (silicon nitride, alumina), polymer (polytetrafluoroethylene) and metal (stainless steel) counterparts. They reported that titanium transferred to steel, polymer, and alumina counterface surfaces; however, when sliding against silicon nitride coatings, Al in the Ti–6Al–4V alloy formed chemical compounds and Ti transferred to the counterface in the form of amorphous titanium oxides.

The findings of Jaffery et al. and Jawaid et al. [\[8,9\]](#page--1-0) revealed that the adhesion tendency of titanium alloys is also responsible for chipping and flaking of the coated cutting tools during machining operations due to the periodic formation and spallation of the titanium transfer layer. They suggested that the low thermal conductivity of titanium alloys was another influential factor for the significant material transfer to the counterparts. Titanium nitride (TiN), aluminum titanium nitride

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(AlTiN) and chromium nitride (CrN) are established wear resistant coatings that are extensively applied on cutting tools in the form of mono- or multiple-layers to improve the performance during machining operations [\[10,11\].](#page--1-0) CrN- and AlTiN-based coatings are extensively applied on cutting tools during machining operations of titanium alloys. CrN coatings are preferred over TiN coatings for unlubricated machining operations since they provide better wear resistance on account of their higher fracture toughness and lower coefficient of friction [\[12,13\].](#page--1-0) In another investigation, Khan et al. [\[14\]](#page--1-0) reported that AlTiN coatings on machining tools resulted in improvements in tool wear and specific cutting energy during machining of titanium-17 alloy. The strong adhesion tendency of titanium alloys and their extensive transfer to various mating materials such as steels, aluminum alloys, ceramic coatings, and polymers under sliding conditions can be minimized by application of surface engineering techniques [\[6,7,15](#page--1-0)–19]. However, the micromechanisms involved in the transfer of titanium to counterface surfaces remain unclear. With the aim of addressing the adhesion problem of Ti alloys to cutting/machining tools, this research focuses on understanding the mechanisms involved in the adhesion and transfer of Ti–6Al–4V to different counterface materials during the initial stages of sliding. In this work, AlTiN and CrN coatings, commonly utilized for bearing applications, as well as AISI 52100 steel were selected as potential mating surfaces for Ti–6Al–4V alloy, and their performance in terms of wear loss and coefficient of friction during both ball-on-disk and single pass sliding tests were evaluated and compared.

2. Materials and experimental procedure

Ti–6Al–4V alloy used in this research was received in the mill-annealed condition with an equiaxed microstructure consisting of primary α grains and β particles at α grain boundaries (Fig. 1). The alloy had an average hardness of 350 \pm 6 HV_{0.05}, measured according to ASTM E384 standard using a Buehler Micromet II microhardness tester at an applied load of 50 grf (0.49 N) and an elastic modulus of 114 GPa [\[20\]](#page--1-0). The surfaces of Ti–6Al–4V disks (25 mm diameter and 5 mm thickness) were polished to an average surface roughness (R_a) of 25 ± 5 nm (measured using optical profilometry) by the conventional metallographic procedure for titanium alloys. The counterface materials were 12 mm-diameter AISI 52100 steel balls coated with CrN and AlTiN coatings. The AlTiN coating had a thickness of 3.5 ± 0.2 µm and the CrN coating had a thickness of 4.5 \pm 0.2 µm. Both coatings were deposited in a commercially available cathodic arc physical vapor deposition (PVD) system.¹ In order to minimize the effect of initial surface topography on the adhesion and friction behavior, the counterface balls were mechanically polished with 3 μm diamond suspension followed by 1 μm diamond suspension on short napped polishing cloth to an average surface roughness (R_a) of 58 \pm 7 nm (measured by optical profilometry). The details of properties and roughness of the counterface balls are summarized in [Table 1.](#page--1-0) The ball samples and Ti–6Al–4V disks were ultrasonically cleaned in an ethanol bath and dried before the experiments.

The ball-on-disk tests were carried out at 3 N load, 0.167 mm/s speed for a total sliding distance of 10 m (200 cycles) using Bruker's UMT tribometer. The amount of wear of Ti–6Al–4V disks was determined by weight measurements before and after each test using a Sartorius LE225D electronic balance to the precision of 0.1 mg and wear rates were obtained by dividing mass loss by the total sliding distance. The single pass sliding tests were also conducted for a sliding distance of 0.01 m at the same sliding speed (0.167 mm/s) and normal load (3 N load) as the ball-on-disk tests to investigate the adhesion behavior of Ti–6Al–4V to different counterface materials.

All the tests were performed in ambient conditions at 25 °C and 40% relative humidity. Each test was repeated three times, and the reported wear rates are the average values obtained from three tests. Real-time dynamic load and tangential force values were recorded during the tests and the coefficient of friction (COF) values were calculated accordingly. The tests were also conducted against an uncoated AISI 52100 steel ball under the same conditions, for the purpose of comparison.

The surface roughness of disks and ball samples were measured before and after the tests using an optical profilometer (Veeco, WYKO). After the tests, the sliding surfaces on Ti–6Al–4V disks and balls were examined under a scanning electron microscope (SEM, Quanta 200 FEG-SEM) equipped with energy dispersive X-ray spectroscopy (EDS). A dual beam Zeiss NVision 40 equipped with a focused ion beam (FIB) milling instrument and a field emission gun (FEG) scanning electron microscope (SEM) was employed for cross-sectional examination and imaging of the microstructure of titanium transfer layers and their adhesion to the counterface balls. Prior to the milling process, a thin (2 μm) tungsten layer was deposited on the surface at regions of interest to minimize the damage during the milling process. Detailed investigation of the microstructure of the transfer layers specifically at the interface region with different counterface surfaces were carried out using a FEI Titan 80-3000 transmission electron microscope (TEM). Elemental analysis of the microstructural features were performed using electron energy loss spectroscopy (EELS) and EDS microanalysis and structural information and phase identifications were performed with high-resolution TEM (HRTEM) imaging and corresponding fast Fourier transform (FFT) patterns as well as convergent beam electron diffraction (CBED) patterns. The TEM samples were prepared using the FIB lift-out technique [\[21\]](#page--1-0).

3. Results

3.1. Ball-on-disk tests

[Fig. 2](#page--1-0) shows the wear rate of Ti–6Al–4V disks against different counterface materials after the ball-on-disk tests determined from mass loss measurements. Similar values of wear rate were obtained when Ti–6Al–4V slides against steel and AlTiN; however, slightly lower wear rate was obtained against CrN. [Fig. 3](#page--1-0) shows the sliding track morphologies on the surface of Ti–6Al–4V disks after the ballon-disk tests against different counterface materials (steel, CrN, and AlTiN). It can be observed that Ti–6Al–4V experienced severe damage after 10-meter sliding tests against all the tested counterface materials.

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Fig. 1. A secondary electron SEM image of the microstructure of the as-received Ti–6Al–4V alloy used in this investigation (etched in Kroll's). The alloy had a "mill-annealed" microstructure consisting of primary equiaxed α grains and grain boundary β particles.

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