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## Investigation on tribo-layers and their function of a titanium alloy during dry sliding



### Q.Y. Zhang, Y. Zhou, L. Wang, X.H. Cui<sup>\*</sup>, S.Q. Wang<sup>\*</sup>

School of Materials Science and Engineering, Jiangsu University, Zhenjiang 212013, China

#### article info

## ABSTRACT

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#### 1. Introduction

Rigney proposed that the sliding wear of metal alloys can be described by the evolution of the following phenomena: surface and subsurface plastic deformation, formation of debris and material transfer, reaction with the environment and mechanical mixing [\[1\].](#page--1-0) Usually, a tribo-layer is formed on worn surfaces. In the most cases, the tribo-layer is considered to be a mechanically mixed layer (MML), which is comprised of materials from both the sliding counterparts  $[2,3]$ . It is clear that the formation of tribolayers is an inevitable phenomenon during the sliding of metals. When tribo-layers, especially different from the metal alloy itself, exist on worn surfaces, the variation of its wear behaviour and mechanism can be expected.

Tribo-oxides inevitably appear in air, thus tribo-layers are generally referred to be tribo-oxide layers. The tribo-oxides are considered to protect from wear in steels, thus oxidative mild wear prevails [\[4](#page--1-0)–[14\].](#page--1-0) Quinn made a great contribution to the research concerning the oxidative wear and tribo-oxide layer in steels [\[4,5\].](#page--1-0) In the subsequent researches, the protective role of tribo-oxides was further confirmed and widely accepted in steels and some nonferrous alloys  $[6-18]$  $[6-18]$ . Due to preventing the adhesion of metal asperities, tribo-oxides protect metal against wear. The wear rate can be reduced by more than one order of magnitude. However, as the metal matrix is thermally softened, tribo-oxide layers lose

E-mail addresses: [cueixianghong@sohu.com](mailto:cueixianghong@sohu.com) (X.H. Cui), [shuqi\\_wang@ujs.edu.cn](mailto:shuqi_wang@ujs.edu.cn) (S.Q. Wang).

<http://dx.doi.org/10.1016/j.triboint.2015.10.018> 0301-679X/© 2015 Elsevier Ltd. All rights reserved. Tribo-layers as mechanically mixed layers were always noticed to form on the titanium alloy; their composition, phase and status changed with sliding conditions. Tribo-layers of the titanium alloy were classified into no-oxide tribo-layer and tribo-oxide layer. It was not tribo-oxide layer, but no-oxide layer that caused poor wear resistance of the titanium alloy. The wear performance of titanium alloys was suggested to be improved by controlling the formation of tribo-oxide layers.  $\odot$  2015 Elsevier Ltd. All rights reserved.

A double-sliding test was performed in a titanium alloy; the tribo-layers and their function were investigated. The tribo-oxide layers of titanium alloy were proved to possess the protective function.

> their protective function because of no firm support from the matrix. In this case, the mild-to-severe wear transition of oxidative wear occurs [\[11](#page--1-0)-[15\].](#page--1-0)

> In titanium alloys, however, the understanding for tribo-oxides seems to be queer; tribo-oxides are widely considered to be of no protection [\[19](#page--1-0)–[26\].](#page--1-0) Straffelini summarized the poor tribological performance of titanium alloys and was mainly attributed to no protective tribo-oxides [\[21](#page--1-0)–[24\].](#page--1-0) This equals to declaring that titanium alloys will never be used as wear resistant parts unless they are modified by coatings [\[19,22\].](#page--1-0) Because of the notoriously poor tribological performance of titanium alloys, the research concerning the wear of titanium alloys themselves has been much limited till now [\[19](#page--1-0)–[36\]](#page--1-0). Oppositely, much work was performed for the wear of titanium alloys with various coatings. Most researchers indeed accepted such a view that tribo-oxides were of no protection in these limited studies on the wear of titanium alloys [\[19](#page--1-0)–[26\]](#page--1-0). For example, Straffelini and Molinari et al. considered that the tribo-oxide layer of Ti-6Al-4V alloy did not adhere to the substrate, was brittle and tended to be continuously fragmented, thus presenting no protective role [\[21](#page--1-0)–[24\]](#page--1-0). Qiu et al. [\[25,26\]](#page--1-0) emphasized a strong correlation between wear behaviour and tribo-oxide layer, and found that the formed oxides of TiO,  $TiO<sub>2</sub>$  and  $V<sub>3</sub>O<sub>4</sub>$ , were very loose and easy to spall off. In addition, in the other studies about the wear of titanium alloys in dry sliding [\[27,28\]](#page--1-0), reciprocation sliding [\[29\]](#page--1-0) and aqueous conditions [\[30\],](#page--1-0) tribo-oxides and their function were not mentioned.

> However, Chelliah and Kailas's research demonstrated that dry sliding wear behaviour of titanium was governed by combination of tribo-oxidation and strain rate response in near surface region [\[31\].](#page--1-0) This meant that tribo-oxides, to some extent, were of

 $*$  Corresponding authors. Tel./fax: +86 5118688797618.

protection function in the dry sliding wear of titanium. But they did not provide the characterization of tribo-oxides and evidence. In our recent work [\[32](#page--1-0)–[35\]](#page--1-0), Ti-6Al-4V and Ti-5.84Al-3.86Mo-1.57Zr-0.32Si alloys were found to be of excellent high temperature wear resistance which was guessed to be resulted from the protective function of tribo-oxides. These seemed to be just contrary to the traditional view of no protective tribo-oxides in titanium alloys [\[19](#page--1-0)–[26\]](#page--1-0).

The above studies aroused a controversy on the protective role of tribo-oxides during the sliding wear of titanium alloys. Clearly, the traditional view "no protective function of tribo-oxides" has been popularly accepted. Although tribo-oxides were considered to be protective in some researches, these are short of the direct evidence [\[31](#page--1-0)–[35\]](#page--1-0). The purpose of the present study is to characterize tribo-layers of titanium alloy and ascertain the protective role of tribo-oxides. The tribo-layers were examined by XRD, SEM and EDS. The specially designed double-sliding test was used to provide the direct evidence to confirm the protective role of tribooxides. The wear mechanisms of the titanium alloy under various sliding conditions were explored as well.

#### 2. Experimental details

A commercial TC11 alloy (Ti-6.5Al-3.5Mo-1.5Zr-0.3Si) was selected to fabricate the flat-ended pins with dimensions of 6 mm in diameter and 12 mm in length. Its chemical composition (wt%) is 5.84Al, 3.86Mo, 1.57Zr, 0.32Si and balance Ti. The titanium alloy was heated at 955 °C for 2 h and water quenched for solid dissolution, subsequently aged at 540 °C for 4 h and cooled in air (36 HRC). A commercial AISI 52100 steel was chosen as counterface discs with dimensions of 70 mm in diameter and 10 mm in thickness. The steel was austenitized at 850 °C, oil quenched, and tempered at 600 °C for 2 h to achieve a hardness of 39 HRC. Through such a heat treatment, the discs retained an unchangeable hardness during sliding, especially at elevated-temperature. The similar hardness of TC11 alloy and AISI 52100 steel was noticed to facilitate tribo-layers to form and stably exist in our previous research [\[34,35\].](#page--1-0) In addition, for the sliding pair with similar hardness, MML formed and contained components from both sliding pin and counterface according to the work of Pau-schitz et al. [\[36\]](#page--1-0). The microstructures of the titanium alloy (equiaxed α particles in an aged β matrix) and steel (tempered sorbite plus dispersive carbide particles) are exhibited in Fig. 1.

Dry sliding wear tests were performed on a pin-on-disc high temperature wear tester (MG-2000 type). The single-sliding tests were conducted at the ambient temperature of 25 and 600 °C, respectively. The sliding distance was selected at  $1.2 \times 10^3$  m (about 20 min of sliding time) and the sliding velocity was controlled at 1 m/s. On the basis of the single-sliding tests, a double-sliding test was specially designed, in which the same specimen and surface first slid at 600  $\degree$ C and then at 25  $\degree$ C. The other parameters in the double-sliding test, such as sliding speed and sliding distance were the same as ones in the single-sliding tests. The first sliding was assumed to form a protective tribo-oxide layer at 600 °C. Conversely, in the second sliding (at room temperature), there was not the formation of tribo-oxides. Therefrom, the tribo-oxide layer was separated as an independent factor, thus its function was revealed through comparing the wear rates of the single-sliding test and the double-sliding test. It must be noted that the wear rate of the double-sliding test was calculated merely from the wear loss in the second sliding at room temperature.

Prior to wear test, the surfaces of the pins and discs were polished with a #400 emery paper and degreased. Each measurement was preceded by an ultrasonic washing of the pins in acetone and subsequently drying. Then, the mass loss was weighed by a balance with an accuracy of 0.01 mg. The worn specimens were firstly cut along the direction parallel to the sliding direction and perpendicular to the worn surface. Then worn surfaces were directly examined by SEM. In order to characterize tribo-layers, worn surfaces were prepared by mounting with resin to investigate cross-sections. The morphology, composition and phase of worn surfaces and subsurfaces were investigated using a scanning electron microscope (SEM), an energy dispersion spectrometer (EDS) and an X-ray diffractometer (XRD) with Cu Kα radiation, respectively. It must be noted that the zone of severe plastic deformation only appeared after sliding. The microhardness of tribo-layer and substrate was measured by a digital microhardness tester with a load of 0.49 N and a hold time of 15 s. The hardness of the alloys was determined by a Rockwell apparatus.

#### 3. Results and analysis

#### 3.1. Characterization of tribo-layers

Three wear tests were performed for the titanium alloy: the single-sliding at room temperature and 600 °C, respectively; the double-sliding with first sliding at 600 °C and subsequently at room temperature. The tribo-layers were distinguished from the matrix through examining the cross-section morphology at worn subsurfaces; the phases and compositions of tribo-layers were identified by XRD analysis of worn surfaces and line scanning at worn subsurfaces, respectively. [Fig. 2](#page--1-0) illustrates the morphology, EDS line analysis and XRD patterns of tribo-layers under various sliding conditions. It was observed that tribo-layers always formed on worn surfaces. Interestingly, the tribo-layers in the latter two conditions presented almost the same morphology, compositions and phases, but differed from the ones in the first condition.



Fig. 1. Microstructures of the titanium alloy (a) and steel (b).

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