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RESEARCH

The Wear behavior of UHMWPE against Surface Modified CP-Titanium by Thermal Oxidation

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A B S T R A C T

The effects of thermal oxidation duration on hardness, roughness, and wettability of the CP-titanium surfaces were investigated in this paper. The thermal oxidation treatment was done at 700 °C for 12-36 hours in an air atmosphere. The wear behavior of the UHMWPE sliding against treated thermal oxidation of the CP-titanium was tested by a pin-on-plate tribometer under lubrication of the solution of 75 % distilled water and 25 % bovine serum. The results showed that the layer of the oxide titanium was formed on the surface after being treated by the thermal oxidation for 12-36 hours. The oxide titanium layer was dominated by rutile form of T_iO_2, that offers an improvement of hardness and wettability of the CP-titanium surfaces. The average wear factor of the UHMWPE reduced significantly when the sliding against of the CP-titanium was modified by the thermal oxidation, and the lowest average wear factor was reached when the sliding against the 12 hour oxidized CP-titanium counterfaces.

1. INTRODUCTION

In general, artificial human joints are composed of at least two components that are arranged, such a manner so that it can function like natural joints. Metallics and polyethylene biomaterial are the most common materials used as a joint implant component. They are combined particularly in the joint implant, that is, one of the components is made of metal, and others of the polyethylene [1]. The most popular type of polymer in the joint implant is called ultra-high molecular weight polyethylene (UHMWPE). It has been widely used as a bearing material in total joint replacement, especially in hip and knee implant, due to its high wear resistance, low friction, and biocompatible. The bearing that made of UHMWPE have excellent performed in vivo. The only major issue is wear and the wear particles on the in vivo durability of the prosthesis [2].

The metallic biomaterials, titanium, stainless steel, and Co Cr are used in the overwhelming majority of the joint implants [3,4]. Recently, titanium and titanium alloys are intensively studied for use in medicine, particularly in dental and orthopedic [5]. It is because the titanium and titanium alloys have higher mechanical properties, lightweight, corrosion-resistant, non-toxic, low modulus, non-magnetic and higher biocompatibility than stainless steel

or cobalt alloy [5,6]. Additionally, they are very reactive metals and will quickly react to form oxide layer when exposed to the atmosphere. The compact oxide layer will form easily at the surface, so that they have excellent corrosion resistance [7–9]. The oxide layer is compact and stable, but the layer which is automatically formed is thin, and it is not enough to improve the wear resistance. All joint prostheses are articulated with a boundary or mixed lubrication that allows the bearing surface in the contact and results in the generation of wear debris [11]. In the total joint replacement, the UHMWPE wear debris is the main issue due to the wear debris is recognized as a leading cause of failure of the total joint replacement [12]. The debris of the wear will cause damage to body tissue surrounding the implant [3,12]. The weakness of the titanium and titanium alloy are poor tribology properties when rubbing against itself or other materials [14]. Therefore, it has limited their extensive use in biomedical implant [13,14].

The one of the methods to improve the tribological properties of titanium is the surface modification. Several studies have been conducted to investigate surface modification of the titanium and titanium alloy material through anodic oxidation, DLC coating, SMAT, and thermal oxidation to improve the wear behavior [13–16]. Thermal oxidation (TO) is very straightforward and inexpensive method. Therefore, this treatment is very promising for improvement of the wear behavior of the titanium which would be used as the load-bearing implant [19,20].

The hard and thick T_iO_2 layers can be obtained on the surface of the CP-titanium by the thermal oxidation method. This method exploits the low resistance of the titanium to oxidation and oxygen diffusion at high temperature. According to previous studies [8,19,21], the two primary parameters to obtain optimum T_iO_2 layer are temperature and duration of the thermal oxidation treatment. Satendra Kumar et al. [23] investigated the formation of rutile T_iO_2 during thermal oxidation of CP-Ti was minimal at 650 ^oC. At the low temperature (below 500 ^oC) the kinetic reaction was slow, but at a high temperature (above 800 ^oC) would affect on the spallation of the oxide layer, so the oxidation temperature was chosen as an optimum temperature between 600 and 700 ^0C [24]. The long duration (more than 72 hours) gave the effect of the spallation of the oxide layer, but if the duration was very low (less than 5 hours) the oxide layer formed was thin [25]. In the previous investigation, the thermal oxidation at 700 ^oC for several duration could reduce the wear of the UHMWPE-CP-titanium/Ti64 counterparts. Wang et al. [26] investigated the wear of Ti64 that was treated by the TO and UHMWPE; they found that the wear volumes decreased 68 % for treated samples compared to the untreated specimen. Xiong et al. [27] investigated the wear properties of the UHMWPE sliding against modified titanium alloys. The friction coefficients of the UHMWPE sliding against thermal oxidation treated specimens under bovine serum that were decreased by 63 %. All studies used the pin on disk tribometer.

A linear reciprocating movement generated on the uni-directional pin on plate tribometer is a more realistic motion for investigating wear mechanism of the joint implant material. The intermittent motion at the end of stroke can represent the natural joint movement [27,28]. The aim of this study was to investigate whether the effect of the duration of the thermal oxidation on the surface treatment of the CP titanium at 700^oC was able to reduce the wear of the UHMWPE tested using a reciprocating pin-on-plate tribometer.

2. MATERIAL AND METHODS

2.1 Materials preparation

The CP-titanium cast was used in this study. The specimens as-received from the investment casting cut by milling machine to the dimension of 55 mm x 18 mm x 3 mm. After that, the specimens were annealed at 900^oC for 2 hours in an argon environment and then cooled in the furnace to obtain a uniform initial microstructure condition. The specimens were ground with abrasive paper #600, 1200, 5000, and finished with diamond paste to obtain average surface roughness (Ra ≈0.1 μm). All of the specimens were ultrasonically cleaned in distilled water for 15 minutes and degreased with alcohol before treatment. In this study, all of the thermal oxidation processes were performed at 700 ^oC.

The oxidation process was performed in a chamber furnace (Thermo Scientific, USA) in an air atmosphere. The specimens were heated in room temperature at 700 ^{o}C with the ramp rate was the default of the furnace setting. The holding time was set 12 and 36 hours (not including the ramp). After the holding time had been reached, the specimens were cooled naturally in the furnace. The specimens were weighed prior and after oxidation with microbalance with an accuracy ±0.00001 g (Ohaus).

2.2 Surface characterizations

The specimen surface roughness was measured using a contact stylus profilometer (Surfcorder, Kosaka, Japan). The measurement was conducted at 19 different locations to obtain the medium arithmetic value (*R*a) of the samples. All samples were cleaned up using alcohol, rinsed in distilled water, and dried prior to the observation and measurement.

The surface morphology of the oxide layer formed in surface CP-titanium was examined using a scanning electron microscope (JSM-6510LA, JEOL, Ltd., Japan). The examinations were conducted on the untreated, 12, and 36 hours treated sample. X-ray diffraction (XRD) was undertaken using Monochromatic X-ray radiation from a copper tube with a wavelength of Kα1 = 1.5406 Å to identify phases presence in the thermally oxidized samples. The X-ray tube was supplied by an electric current at a voltage of = 40 kV and intensity of I = 30 mA. The scanning was done in an angular range from 10^{o} to 90^{o}.

The surface hardness examinations were determined using a Vickers microhardness tester (Buehler, USA). The tests were conducted with indentation load 0.49 N for 15 s. The measurement of the average hardness was done for seven different locations at the surface.

The surface wettability was taken from the measurements of the drop static contact angle between the bovine serum solution (75 % distilled water and 25 % bovine) and sample surface at room temperature. A bovine solution droplet was deposited five times at three different locations on the surface of each sample. The droplet of the bovine solution image was obtained using a USB digital microscope attached in water drop equipment (Gaosuo, China) and then the droplet contact angles were analyzed.

2.3 Unidirectional reciprocating wear test

The wear test was performed for untreated and the oxidized CP-titanium specimen with the UHMWPE in the 6 station unidirectional reciprocating pin-on-plate tribometer. The device was built in our laboratory. The flat-ended UHMWPE pins having the dimension of the Ø 9 mm, and 15 mm length were prepared as counterpart specimen. Prior to wear testing, all the UHMWPE pin specimens were allowed to soak in distilled water for 2 weeks to prevent the moisture uptake during the wear test. The density of the UHMWPE was assumed 0.9737 gr/cm^3.

The UHMWPE pins were installed at the upper of the plate sample moving unidirectional reciprocating. The normal load on the pin was 180 N which was provided with the lever mechanism, so that it corresponded to 2.8 MPa of nominal contact stress. The frequency for reciprocating motion was set 1 Hz and the sliding distance was set to 25 mm giving a sliding velocity of 50 mm/s. The wear tests were done at a room temperature. The lubricant used in this study was a solution of 25 % bovine serum and 75 % of distilled water. To prevent the lubricant from the bacterial growth the sodium azide were added (0.1 % v/v).

The wear tests were run with a total sliding distance of about 75 kms. The tests were interrupted at regular intervals 12.5 kms and the wear was measured by the weight loss. The wear device was shut off then the pins and plates were removed for weighing every regular interruption. Prior to weighing, the pins and plates were cleaned with an ultrasonic cleaner in distilled water for 15 minutes and lapped with tissue.

The samples were weighed using a microbalance with an accuracy ±0.00001 g (Ohaus). The average wear factor was calculated as accumulated volume loss (mm^3), divided by the unit of force (N) times total sliding distance (m). The average wear factor (WF) was determined from Eq (1):

$$WF = \frac{Wear\ Volume\ (mm^3)}{Load\ (N)\ x\ sliding\ dist\ (m)} \quad (1)$$

3. RESULT AND DISCUSSION

3.1 Microstructure initial state

Microscopic metallographic examinations showed that the CP-titanium cast after being annealed at 900 °C for 2 hours was characterized by a grain structure with the equiaxial grains shape (Fig. 1).

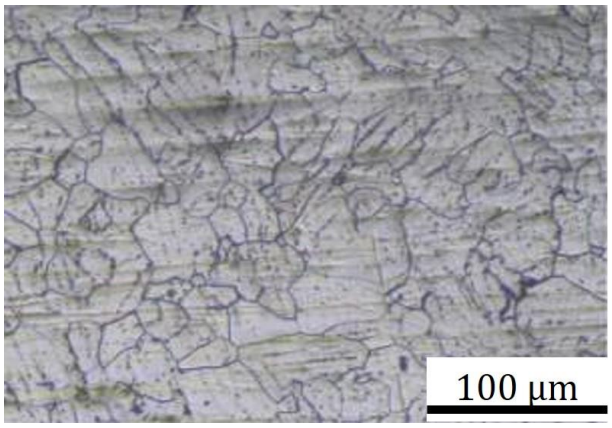

Fig. 1. The microstructure CP-titanium cast after annealed at 900 °C for 2 hours.

3.2 Surface morphology

Figure 2 shows the diffractogram obtained for CP-titanium samples untreated and TO-treated at 700 °C for 36, 12 hours. According to JCPDS card (No. 88-1175), the peaks at diffraction angle 2θ = 27°, 36° , and 55° indicating T_iO_2 in rutile phase [30]. Thermal oxidation treatment samples showed peaks as the rutile phase were dominant. This condition means that the oxidized surface is mainly composed by the rutile form of T_iO_2. Increasing the duration of the treatment, the composition of the oxide layer would be dominated by rutile T_iO_2. It has been reported that the thermal oxidation of pure titanium at high temperature (650-700 °C) and for short times to obtain to the formation of rutile T_iO_2 [8,23,28]. The oxidation temperature and duration of treatment employed in this study were sufficient for the formation of the rutile oxide. As known, rutile phase is a stable structure of T_iO_2 with high hardness so the thermal oxidation treatment of the CP-titanium would improve the wear behavior [10]. The SEM images of the surface sample for untreated and treated at 700 °C for 12 and 36 hours are shown in Fig. 3. The surface of the sample of the CP-titanium that was treated at the temperature 700 °C revealed the existence of a uniform oxide layer which covered the entire of the sample surface.

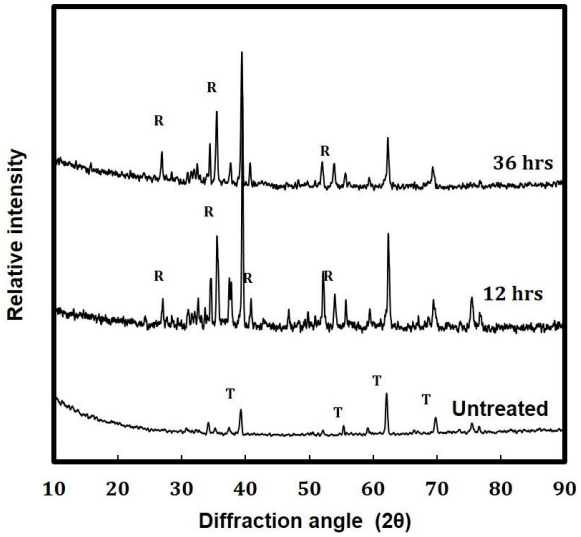

Fig. 2. XRD patterns of untreated and thermal oxidation of CP-titanium at 700 °C for 12 and 36 hours.

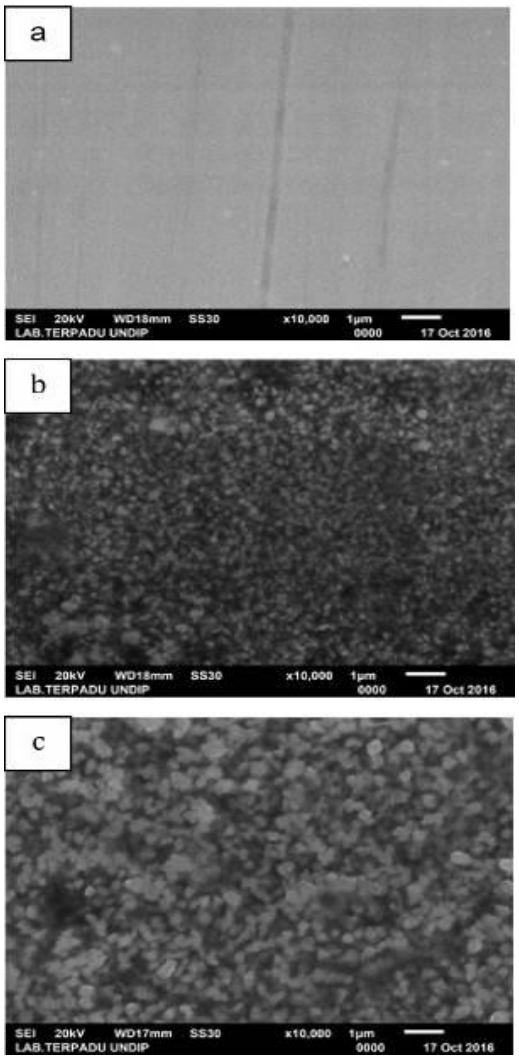

Fig. 3. The SEM image of the surface sample, (a) untreated, (b) treated at 700 °C for 12 and (c) treated at 700 °C for 36 hours.

After 12 hours of the oxidation, the oxide layer covers throughout the surface (Fig. 3b). The grains of the oxides formed were noticeably larger after being treated at 700 ◦C for 36 hours than 12 hours. The oxide layer was relatively smooth for a sample oxidized for 12 hours but it would be relatively coarse when oxidation time was increased to 36 h. According to the literature [32], the formation of an oxide layer on the surface of pure metal were done in four steps, first, oxygen adsorption at the surface, second, the formation of oxide nucleation, third, lateral growth of the nuclei and the last, formation of a compact oxide layer. When the lateral growth of the nuclei is terminated, the metal surface is completely covered by the oxide layer.

From the Fig. 3, It can be seen that the oxide layer grows through the formation and agglomeration that become very fine grains of oxide particles (Fig. 3b). Along with increasing time of the treatment (36 hours), the oxide particles would grow to be larger (Fig. 3c). This is similar to the study of the growth of the oxide layer was found in a paper [33].

Figure 4 shows the surface microhardness of untreated and thermal oxidation treated samples measured on the surface of the oxide layer. The surface hardness of the treated CP-titanium at 700 ^{0}C for 36 hours (Hv = 8.3 GPa) was approximately three times higher than the surface hardness of the untreated CP-titanium (Hv = 2.78 GPa). It can be seen that the hardness enhances by the increased duration of the treatment. The thermal oxidation conducted for only 12 h at a temperature of 700 ◦C made it possible to obtain an oxide layer almost thrice harder than the untreated material. But in the longer duration of treatment no increase of hardness that did not significantly increased. This is similar to the studies that reveal the increase of the duration of the treatment would increase the microhardness [33]. In addition, the surface roughness also increased by the increase of the duration of the thermal oxidation treatment (Fig. 5). Compared with the untreated sample, the surface roughness of the treated sample increased significantly about two times. The increase of the surface roughness was consistent with the papers [20,23,33]. Surface roughness before the thermal oxidation (*R*a = 0.1 μm) increased to 0.26 μm and 0.30 μm in the thermal oxidation at 700 ◦C for 12 and 36 hours, respectively.

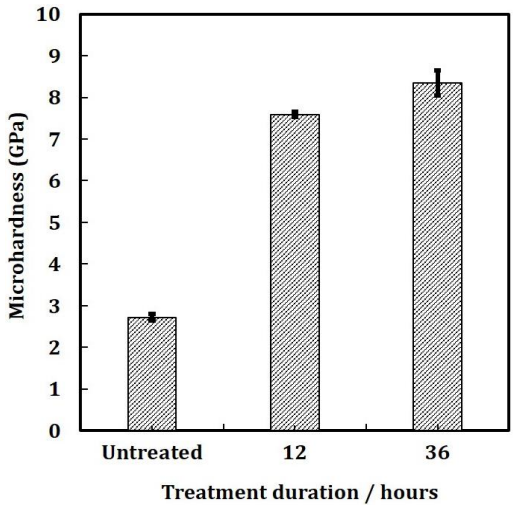

Fig. 4 The surface microhardness of untreated and treated CP-titanium.

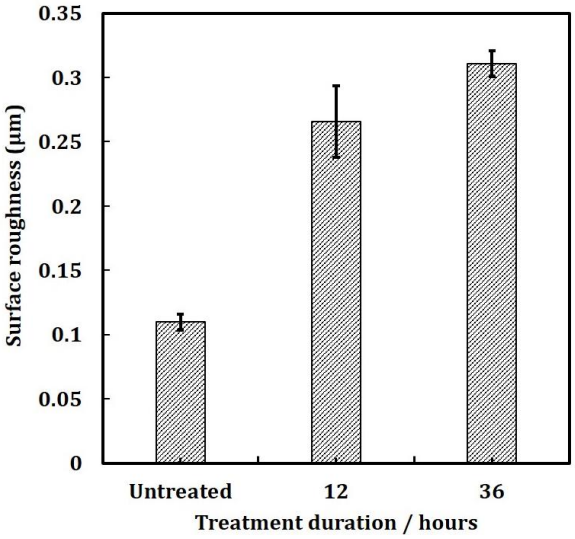

Fig. 5 The surface roughness of untreated and treated CP-titanium.

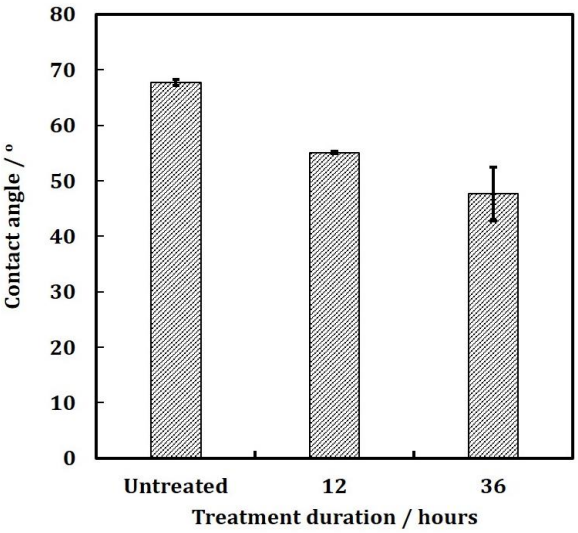

Fig. 6 The static contact angle of untreated and treated CP-titanium.

The increase of the surface roughness was caused by the growth of the oxide layer on the surface. This can be described by the formation of large agglomerations oxide grains on the surface. Grains of the oxide began to cover the surface and grow larger in size when the duration increased (Fig. 3).

The effect of the thermal oxidation duration in the contact angle is shown in Fig. 6. The static contact angle of the untreated sample was about 67°. However, the contact angle of treated sample for 12 and 36 hours were lower than that of the untreated sample. The decrease was about 12-20°. The lower static contact angle means the wettability of the surface improves. This is in good agreement with the studies that reveal the increase surface roughness would reduce static contact angle [35]. The good wettability or low contact angle means the lubricant covered the sample surface easily and it potentially reduced the friction and wear. The film of lubricant that covers the surface will prevent the surface from rubbing each other.

3.3 Tribological test

The graphic of the volume loss compared sliding distance, for the UHMWPE sample against untreated and treated thermal oxidation of the CP-titanium plate as shown in Fig. 7. It is obvious by comparing untreated and treated sample that the wear of the UHMWPE sample was significantly reduced by treated thermal oxidation of the CP-titanium counterface irrespectively with the duration of the treatment.

The average wear factor for the UHMWPE pins against untreated and thermal oxidation CP-titanium are shown in Fig. 8. It was noticed that the wear factor decreased significantly from 1.25 x 10^{-7} mm^3/N m to 3.54 x 10^{-8} mm^3/N m and 5.87 x 10^{-8} mm^3/N m after the thermal oxidation for 12 and 36 hours, respectively. The CP-titanium sample treated for 12 hours showed the fewest wear of the UHMWPE compared with the 36 hours treated and untreated. The average wear factor of the UHMWPE pins against 12 hours and 36 hours thermal oxidation treated samples were reduced 3.5 and 2 times that of the UHMWPE pins against untreated CP-titanium respectively. Although the samples subjected to thermal oxidation had a higher surface roughness than the untreated samples, however, the oxide layer formed on the surface of the samples were able to reduce the wear of the UHMWPE. Specifically for couples the UHMWPE and CP-titanium-treated, showed that the surface roughness also affected the wear though it was not significant.

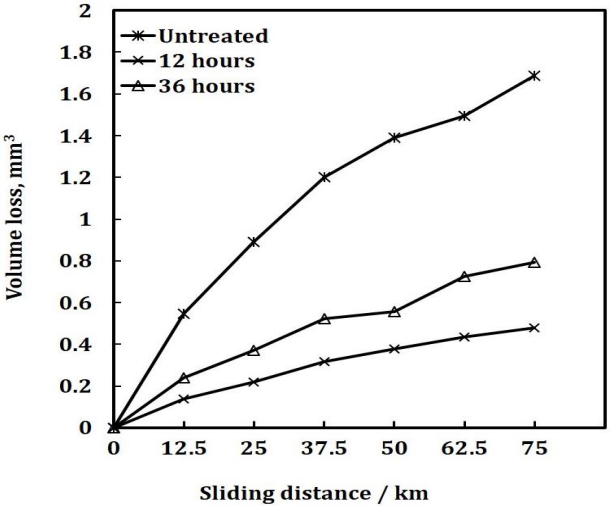

Fig. 7 Effects of thermal oxidation duration of CP-titanium counterfaces on the wear of UHMWPE.

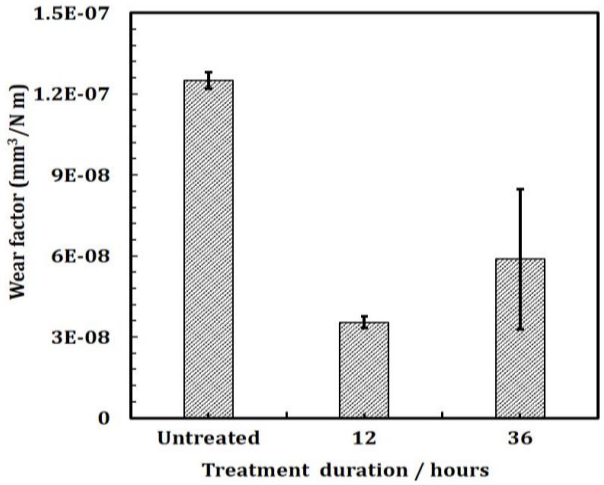

Fig. 8 Average wear factor of UHMWPE sliding against untreated and treated CP-titanium.

The figures of the worn surfaces of the CP-titanium plate and pin tested under bovine serum as lubricant are shown in Fig. 9. The wear track of the untreated CP-titanium plate (Fig. 9a) was marked by relatively deep and wide grooves. Thus is an indication of severe abrasive wear. This matched with the UHMWPE counterface also experiences severe damage marked by several grooves (Fig. 9b). The different wear track can be seen in Figs. 9c-9f, which found only a few scratches on the surface of the treated CP-titanium and UHMWPE surface. This indicates

that the thermal oxidation treatment can improve wettability and reduce wear.

The surface of the CP-titanium was covered by the oxide layer composed of T_iO_2 oxide which improved hardness and wears resistance. The formation of T_iO_2 can be held responsible for an increase the surface roughness and enhance in the wettability. The coarse grain oxide will offer greater surface contact and provide micro spaces between grains (Fig. 3) that play as lubricants pocket to increase hydrodynamic lubrication which results in a decrease of wear rate. The thermal oxidation treatment can be suitable to reduce the wear of corresponding UHMWPE and hinder the generation of abrasive particles. In the case of the untreated CP-titanium counterfaces, although with smooth surfaces, the average wear factor is higher than the treated sample, because the auto oxide layer is thin so that it can not inhibit the wear sliding. The thin oxide layer will peel off as wear debris or will penetrate in the UHMWPE and lead to scratching of the CP-titanium surface which, in turn, leads to severe abrasive wear to both articulate surfaces. The wear mechanism of the UHMWPE/untreated CP-titanium couple may be attributed to the mechanisms of third body wear. This study has shown that the wear of the UHMWPE was greater against CP-titanium plate treated for 36 hours than against 12 hours treated plates, due to the roughening by thermal oxidation duration. In the case of the rough CP-titanium thermal oxidation treated surface, the coarse of oxide grains accelerated the wear because of the high point or asperities on the surface to produce wear by an abrasive wear mechanism.

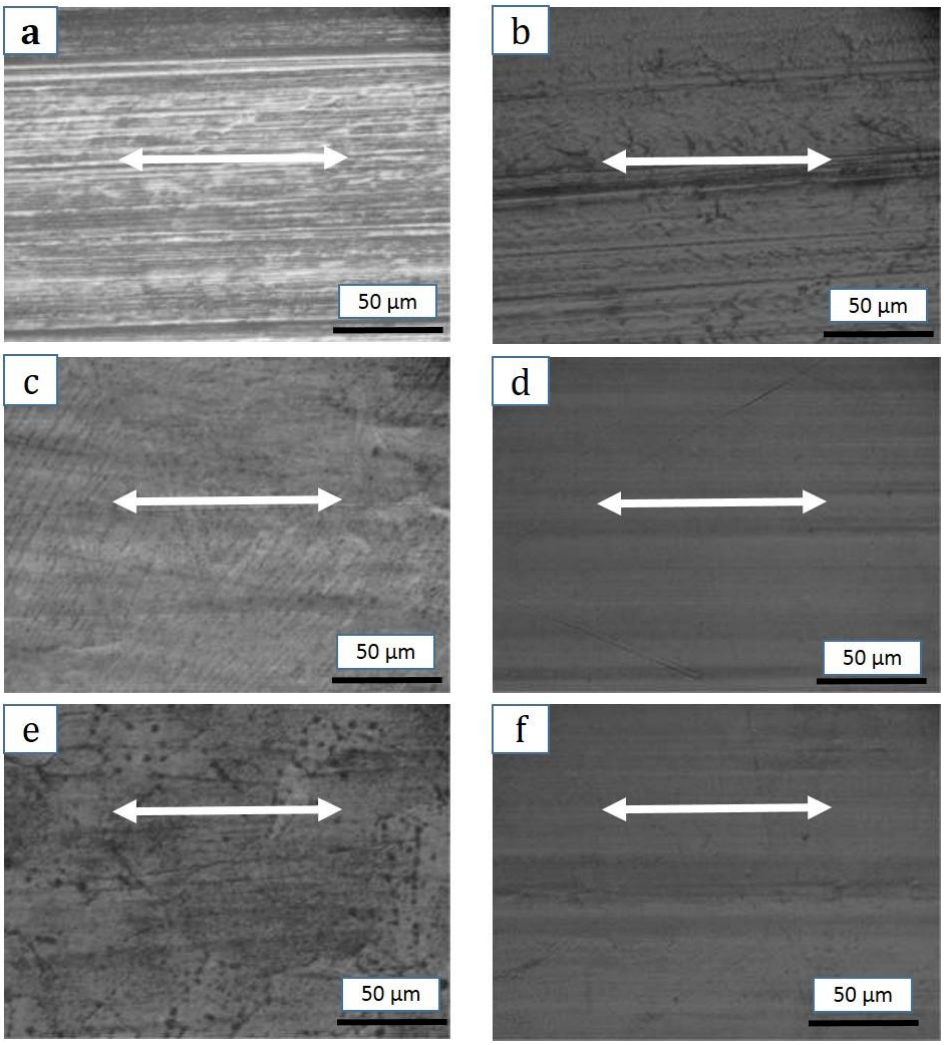

Fig. 9. Worn surfaces of plate and pins (a) untreated CP-titanium; (b) UHMWPE sliding against untreated CP-titanium; (c) 12 hours thermal oxidation CP-titanium; (d) UHMWPE sliding against 12 hours thermal oxidation CP-titanium; (e) 36 hours thermal oxidation CP-titanium; (f)) UHMWPE sliding against 36 hours thermal oxidation CP-titanium. The arrows indicate as sliding direction.

4. CONCLUSION

The rutile T_iO_2 layer was formed on the surface of the CP-titanium after the thermal oxidation treatment at 700 ^oC in an air atmosphere. The surface microhardness of the treated CP-titanium for 36 hours was 8.3 GPa that was three times higher than that of the untreated CP-titanium. The thermal oxidation treatment of the CP-titanium could reduce the UHMWPE average wear factor with the reduction of 71.6 % and 52.9 % for 12 and 36 hours, respectively. The biotribological test revealed that the thermal oxidized CP-titanium decreased the wear of the UHMWPE, which indicated that the thermal oxidation treatment of the CP-titanium could be a potential candidate of the surface treatment for artificial joints.

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