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DEVELOPMENT OF MICRO-HARDNESS AND WEAR PERFORMANCES OF BIOMEDICAL TITANIUM

Mohsin T. Mohammed[1]

[1]Nanotechnology and Advanced Materials Research Unit, Faculty of Engineering, University of Kufa, Najaf, Iraq; Email: mohsint.alyasiri@uokufa.edu.iq

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ABSTRACT

In the present work, the influence of thermal oxidation (TO) process on the micro-hardness and wear performances of a pure titanium (Ti) was studied. The process has been carried out at various values of temperature for determined time followed by furnace cooling to room temperature. The TO has supported the development of strong surface film on the Ti without spallation. This surface structure improved the results of micro-hardness and wear compared to untreated samples.

KEYWORDS: Ti, Surface modification, Micro-hardness, Wear, Biomaterials

1. INTRODUCTION

Ti and its materials are extensively employed in the implantology field for various biomedical applications (Niinomi et al., 2012; Yang, et al., 2013) due to their low density, excellent biocompatibility, in addition to well-established corrosion and mechanical properties (Eisenbarth et al., 2004; Mohsin et al., 2014). Recently, high biocompatible Ti offers an appropriate choice for biomedical load-bearing applications (Abdel-Hady and Niinomi, 2013; Mohsin et al., 2014).

It is well known that Ti rapidly forms a very thin surface oxide layer as soon as exposed to oxygen, consisted of mainly TiO_2. This surface layer provides Ti alloys chemical inertness as well as high wear and corrosion resistances in a biological fluid (Liu et al., 2004). However, this passive oxide layer has poor mechanical performance and may be ruptured at extremely low shear stresses (Lilley et al., 1992). Typically, implantable materials are experienced to various intricate stresses, such as tension, compression, torsion and bending throughout the routine activities, hence, high hardness is essentially required (Mohsin et al., 2015). Furthermore, fretting and sliding wear are considered as main reasons of the degradation and fracture of the passive surface layer (Hoeppner and Chandrasekaran, 1994; Rabbe et al., 1994; Zhu et al., 2009). Undesirable tissue reactions might be existed as a result of the metallic ions and wear debris released throughout the fracture of the passive layer. Unfortunately, Ti has poor tribological properties comparing with other biomaterials. The large variation of tribological properties between the bone and implantable material should be reduced to enhance the service life of the implant and also to keep the bone away from damage (Capitanu et al., 2008). Hence, there was considerable attention from specialist and researchers to improve the friction and wear properties of Ti so as to increase its life span.

There are many surface modification techniques to improve the mechanical and wear properties of implantable Ti, such as sol–gel, plasma spray deposition, physical vapour deposition, chemical vapour deposition, ion implantation, anodic oxidation, thermal oxidation and others. Thermal oxidation is a simple technique used to produce a thick, stable and highly crystalline oxide layer (rutile) on the surface of Ti. In the present research, TO was made at different temperatures to enhance the micro-hardness and wear properties of pure Ti for medical purposes.

2. MATERIALS AND METHODS

Multi Samples with dimensions (1 x 1 x 0.3 cm^3) of commercial pure titanium (CP-Ti), grade No. 2, were used as the base material of TO. Standard techniques of grinding, polishing and cleaning have been used in order to prepare the samples for TO. The conditions of TO, such as temperature, time and atmosphere, were selected to be 500 and 800 °C at heating rate 5 °C /min in air for 8 h as a fixed time. Finally, furnace cooling was utilized to cool the treated samples to room temperature in order to provide homogeneous oxide film on the surface without any defects.

Field emission scanning electron microscope (FESEM, TESCAN MIRA3) was employed to test the oxide film of the investigated samples. The Vickers micro-hardness values of at least five samples were determined by micro-hardness tester (Modele: HVS 1000, Spain), using 200 gf and 10 seconds for load and dwell time, respectively.

Dry sliding wear test of the investigated CP-Ti samples was carried out at ambient temperature on a reciprocating pin-on-disc sliding type wear tester. The wear test parameters of sliding distance and sliding speed were selected to be 5 mm and 10 mm.s-1 respectively. The wear resistance of Ti samples was characterized using the weight loss way and the coefficient of friction according to the recorded friction force and the applied normal load.

3. RESULTS AND DISCUSSION

By visual assessment, diverse colors on the surface of the oxidized Ti samples, from light blue to dark brown, have been observed with rising temperature from 500 to 800 °C. This indicates the growing in the thickness of the surface film formed. As elevated temperature, long duration and high cooling rate through TO may lead to debonding and spallation of the oxide film (Kumar et al., 2010; Jamesh et al., 2013); thus, TO was made in this work in appropriate conditions of those main parameters. The spallation can be considered as a normal result of the large thermal stresses formed due to the difference in the coefficients of the thermal expansion of Ti and its oxide film (Jamesh et al., 2013).

The surface morphology of the oxidized Ti samples at various temperatures (500 and 800 °C) for 8 h is shown in Fig. 1. It can be seen from this figure the creation of oxide film apparent at the surface without spallation. A thin oxide film at the surface along with few nodular particles has been developed during the oxidation treatment at 500 °C (Fig. 1a). On the other hand, the oxidation treatment at 800 °C showed completely covered oxide film (Fig. 1b). As is well known, the direct contact between the surface and oxygen induces the instantaneous nucleation of oxide film all over the surface. Afterwards, two leading mechanisms, growth and agglomeration of oxide particles, take place throughout increasing the temperature from 500 to 800 °C which in turn enhance the porosity of the oxide film.

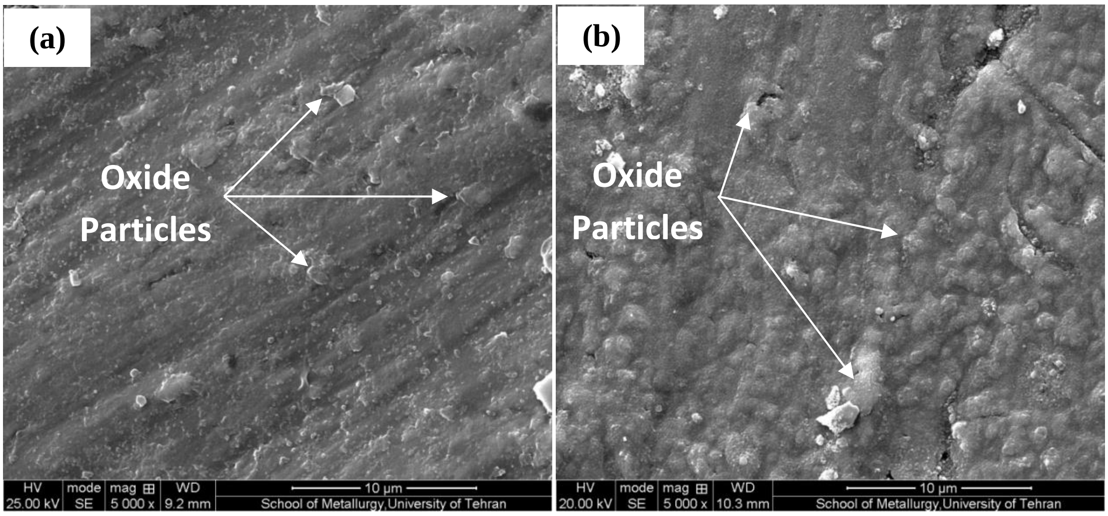

Fig. 1. The morphology of surface treated Ti samples at (a) 500 ◦C and (b) 800 °C.

It has been pointed out in literature that (Ti(O) and rutile (TiO_2) are the major phases in the thin film formed through TO at 500 and 800 °C, respectively (Guleryuz and Cimenoglu, 2005; Siva et al., 2007; Jamesh et al., 2012). The presence of rutile phase in the thin film of the samples treated at 800 °C refers to the improvement of the thickness of the oxide film, while the formation of a thin oxide layer can be indicated from the presence of Ti(O) phase in the thin film of the samples treated at 500 °C (Kumar et al., 2010).

As is well known, Ti oxide is considered as a crucial factor that affects significantly the values of hardness and other properties of biomedical Ti. Therefore, in the present work, the oxidation behavior of the CP-Ti was investigated over two temperatures (500 °C and 800 °C), for 8 h in air. Fig. 2 shows the results of the micro-hardness of oxidized Ti samples in comparison to untreated samples under determined temperatures. The figure obviously reveals the effect of formation oxide film on the results of micro-hardness as these results were significantly higher in treated samples compared to untreated. This considerable enhancement in surface hardness of treated samples is due to the dissolution of oxygen which causes high amounts of strains in matrix with an increase in c/a ratio, in addition to induce great distortion in lattice (Yan and Wang, 2004). The diffusion of oxygen atoms through the surface of Ti samples (oxidation rate) can be improved by increasing the temperature of the TO process. Moreover, the thickness growth of the oxide film throughout the increasing of the TO temperature is also as a result of dissolved oxygen underneath the oxide film (Borgioli et al., 2001). Hence, in the present work, the optimal result of hardness is achieved (HV=524) when the TO was done at temperature 800 °C for 8 h. This higher result of hardness supports the fact that TO is an applicable process for developing the surface characteristics of CP-Ti samples.

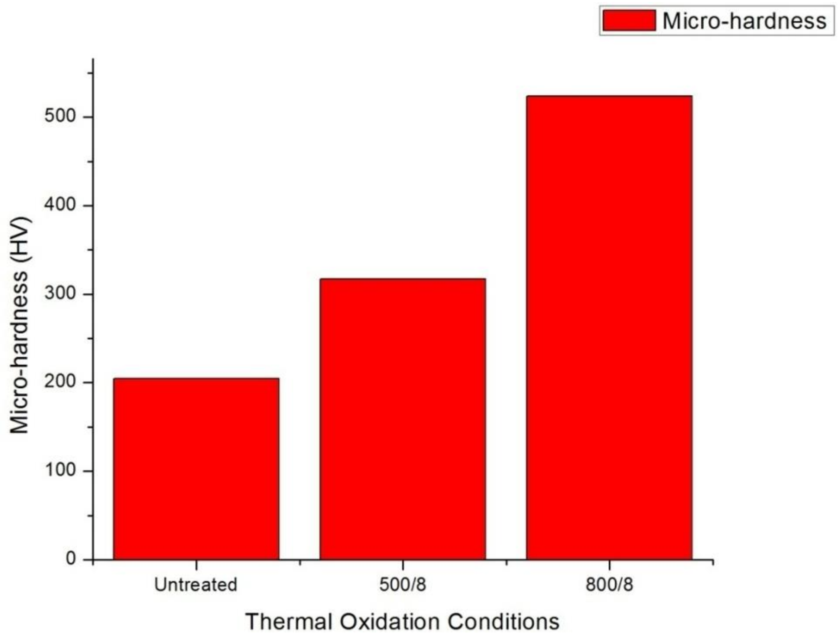

Fig. 2. Surface hardness of untreated, treated at 500 °C for 8 h (500/8) and treated at 800 °C for 8 h (800/8) Ti samples.

Wear loss of untreated and oxidized samples, along with the maximum friction coefficient values, is plotted in Fig. 3. The maximum friction coefficient values followed similar trends to wear loss figure; they were higher in untreated samples than oxidized samples. In general, the friction coefficient and weight loss of oxidized samples at 500 ^{o}C and 800 ^{o}C are lower than that of untreated samples, so that a substantial improvement of the wear resistance is obtained. It can be seen from Fig.3 that the untreated samples underwent significantly more friction coefficient and wear loss than the oxidized samples. Moreover, the friction coefficient and wear loss of samples treated at 500 ^{o}C were lower than in untreated samples. This is may be due to the formed layer of TiO_2 which is created by TO at this temperature. On the other hand, increasing temperature to 800 ^{o}C led to a dramatic decreasing in friction coefficient and wear loss due to the increasing in the thickness of the inner diffusion layer. The changing in the temperatures of TO may produce different oxide and layer thicknesses. In other words, the temperature parameter plays a leading role in drastically increasing the thickness of the oxide film. Thus, the TO at 800 ^{o}C provided an excellent protection against wear.

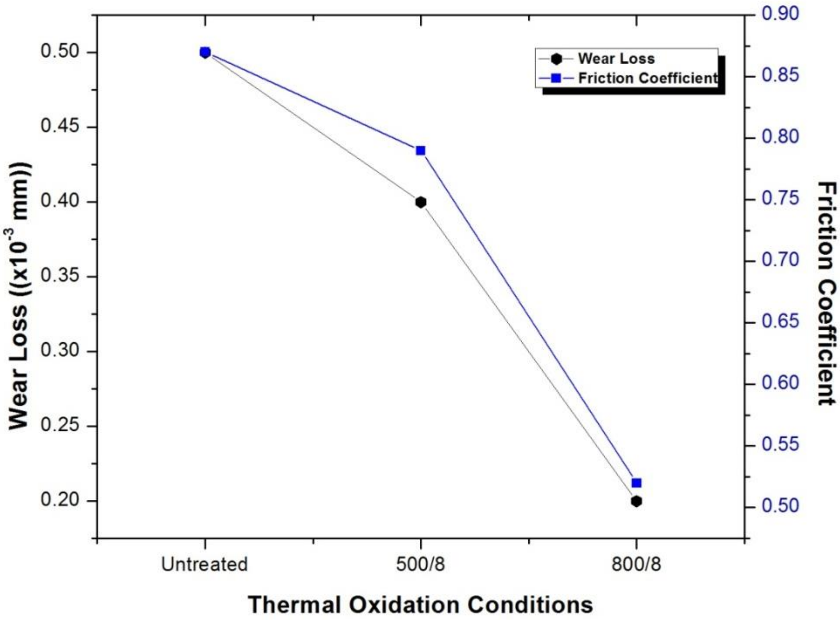

Fig. 3. Wear loss and maximum friction coefficient of investigated CP-Ti samples.

4. CONCLUSIONS

Micro-hardness and wear performances of untreated and thermally oxidized Ti samples at 500 and 800 ^{o}C for 8 h followed by furnace cooling were evaluated for biomedical applications. The following conclusions can be drawn:

1. Both thermal oxidation treatments, at 500 °C and 800 °C for 8 h, lead to form surface oxide scales without spallation.
2. The formation of oxide layer formed on the samples oxidized at 800 °C is more compact and protective than the samples oxidized at 500 °C.
3. TO process at 800 °C for 8 h introduces higher surface hardness in comparison to same process at 500 °C.
4. Oxidation treatment of CP-Ti samples greatly decreases their friction coefficient and wear loss, owing to the formation of a hard, oxide layer on the surface of Ti samples.
5. The oxidation treatment at 500 and 800 °C for 8 h produces enhanced hardness and wear performances of CP-Ti samples compared to untreated Ti samples.

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