Development of a Low-Cost Ball-on-Flat Linear Reciprocating Apparatus: Test Validation Using Ti-6Al-4V and Ti-6Al-4V/Nb₂O₅ Coatings

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Abstract: This work aims to propose the development of a low-cost ball-on-flat linear reciprocating apparatus that can have the same attributes of extremely expensive equipment available on the market. For this purpose, the device was constructed following the recommendations of the ASTM G133 standard. The device's validation was performed trough a comparative study between the ASTM G133 standard and 3D profilometry technique by using Ti-6Al-4V and Ti-6Al-4V/Nb2O5 specimens. The results obtained in the present work were very motivating by showing it was possible to obtain the wear properties with reproducibility through the equipment constructed. The wear tests showed that the functionalization of the Ti-6Al-4V alloy with Nb₂O₅ coatings increased the wear resistance of the biomaterial, reducing the wear volume about 80.1 % when compared to the base material. Finally, this work demonstrated (i) the effectiveness and reproducibility of the device built to carry out microwear tests, (ii) the increase in resistance to the mechanical wear process conferred by the Nb₂O₅ film on the Ti-6Al-4V alloy and (iii) the powerful impact of the 3D profilometry technique when compared to the ASTM G133 standard for wear volume determination.

Keywords: Titanium alloy, Ti-6Al-4V, Thin film, Nb₂O₅, Micro-abrasive wear test.



Graphical Abstract

1. INTRODUCTION

The industrial sector is constantly searching for the development of new methodologies that avoid failures

*Address correspondence to this author at the Institute of Exact Sciences, Naturals and Education, Federal University of Triångulo Mineiro (UFTM). Avenida Doutor Randolfo Borges Júnior, Univerdecidade, 38064200 -Uberaba, Minas Gerais, Brazil; Tel: +55 34 98407 2300; E-mail: jeferson.moreto.uftm@gmail.com of its components. Most of the works related to the micro-abrasive wear of metallic materials are due to titanium alloys, evidencing its importance in the automotive, aerospace, nuclear, chemical, marine, and biomedical sectors [1]. Although titanium alloys exhibit good biological and corrosive properties, their usage is limited to load bearing applications due to their poor tribological characteristics [2]. Research regarding the development and application of niobium pentoxide

 (Nb_2O_5) thin films on the titanium, aluminium and stainless-steel alloys has been performed by this research group in the last years [3-8]. Despite these excellent results, there is a lack of research that considers the effect of Nb₂O₅ thin films on the wear properties of the Ti-6AI-4V alloy. Here, we propose the development of a low-cost ball-on-flat linear reciprocating apparatus to perform microabrasive wear tests following the recommendations of the ASTM G133 [9]. Seeking to validate the designed device, the resistance to wear process was verified by using coated and uncoated Ti-6Al-4V alloy with Nb₂O₅. Its development brings new insights into the reactive sputtering technique to obtain Nb₂O₅ thin films on the Ti-6AI-4V alloy as well as its impact on wear properties of the biomaterial. Consequently, a range of possibilities opens to be explored in the search for obtaining a material that can be effectively used for biomedical purposes.

2. EXPERIMENTAL

2.1. Material

Substrates of Ti-6Al-4V alloy (1.0 cm x 1.0 cm) were used to improve the wear resistance through Nb_2O_5 coatings. Before the deposition process, all substrates were polished with SiC grit papers in the sequence of 800, 1200, 2400 and 4000 mesh. After polishing, the samples were washed in distilled water and isopropyl alcohol by using an ultrasound for a period of 10 min.

2.2. Deposition Process by Using Reactive Sputtering Technique

The Nb₂O₅ thin films were deposited on the Ti-6Al-4V alloy surface by using a reactive sputtering technique. The deposition system is composed by a magnetron sputtering purchased from Kurt Lesker Co. for targets of 2.0 inches diameter. The vacuum is supported by rotative and turbomolecular pumps reaching a pressure of 0.001 mTorr. Gases were admitted and controlled by needle valves from Edwards Co. The film depositions were conducted at 5.0 mTorr with Argon (99.999% White Martins). To obtain Nb₂O₅ stoichiometry films O₂ (99.999% White Martins) at 0.5 mTorr was used. DC voltage and current were set at 450 V and 145 mA, respectively. For these conditions, the thickness of Nb₂O₅ films reached ~400 to 450 nm after 40 min of deposition. The films were shown to be homogeneous from the optical point of view over the entire length of the Ti-6Al-4V substrates without signs of cracking or detachment. Figure 1 shows a schematic drawing of the sputtering system used in the present work.

2.3. Micro-abrasive Wear Tests

The low-cost ball-on-flat linear reciprocating apparatus was developed in the present work following the recommendations of ASMT G133 [9] and validated with the determination of wear volume of uncoated and coated Ti-6Al-4V alloy with Nb₂O₅ thin films. The micro-



Figure 1: Schematic drawing of the sputtering system used to deposit Nb_2O_5 coatings on the Ti-6Al-4V alloy in the present work.

abrasive wear tests were performed on mentioned specimens by using a normal load of 0.5 N at two different time intervals (300 and 600 s), frequency of 0.83 Hz, relative humidity between 40 and 60%, and room temperature. After the micro-abrasive wear tests, the flat samples were cleaned with air jet and conditioned in appropriate sample holders for profilometry analysis. The wear volume ($W_{v,flat}$) was determined by multiplying the average of three cross-sectional areas ($W_{q,avg}$) and the length of the wear track (S) (see Equation 1).

$$W_{v,flat} = W_{a,avg} \times S \tag{1}$$

Figure **2** shows a schematic representation of the flat specimen wear volume calculation according to the ASTM G133 standard [9]. An idealized wear track representation is shown in Figure **2(a)**, whilst a segmentation of the wear track is presented in Figure **2(b)**. The cross-sectional wear profile used to determine the wear volume may be seen in Figure **2(c)**.

2.4. Determination of Wear Volumes by Using 3D Profilometry Technique and ASTM -G133 Standard

As mentioned by Ayerdi [10], the method of obtaining the wear volume of ASTM G133 [6] has some limitations. ASTM G133 standard [9] disregards the rounded areas of the wear track, also known as motion reversal points [11] (See Figure **3**). The omission of this volume added to the irregularity of the track is able to express variations of up to -17.8% in irregular tracks

and -12.5% in regular tracks when compared to the 3D profilometry technique [6]. As the 3D profilometry technique exhibits a good accuracy [12, 13], a comparison of the wear volumes between the two methodologies was performed. For this purpose, the Taylor Hobson white light interferometer equipment (Talysurf CCI Lite M12-3993-03) was used to measure the surface roughness of the coated and uncoated Ti-6Al-4V alloy.



Figure 3: Schematic drawing of the wear track segmentation, demonstrating the central section as well as the round side areas (blue arrows).

AFM analyzes were obtained on Ti-6Al-4V and Ti-6Al-4V/Nb $_2O_5$ samples before the microwear tests by using a Shimadzu SPM9700 microscopy in the



Figure 2: A schematic representation of the flat specimen wear volume calculation according to the ASTM G133 standard [9].

dynamic mode at the Federal University of Triângulo Mineiro (UFTM), Uberaba, Minas Gerais state, Brazil. The cantilevers were purchased from NT-MDT Co. Scanning electron microscopy (SEM) was also performed before and after the micro-abrasive wear tests. For this purpose, an analytical electron microscope FEG-SEM JEOL 7001 F equipped with an Oxford EDX light element detector was used. All analyzes were performed in the Chemistry Department of the Federal University of Uberlândia (UFU), Uberlândia, Minas Gerais state, Brazil.

3. RESULTS & DISCUSSION

3.1. Construction of Microwear Device

A schematic drawing of the device designed and adapted from reference [6] is shown in Figure 4(a). As can be seen, a normal load is applied to the support (1) where a sphere of radius R = 4.75 mm is attached (2), which promotes a relative motion between the sphere



Figure 4: (a) Schematic drawing of the abrasive microwear device based on ASTM G-133 [6] and **(b)** the assembled and finished equipment for performing microwear tests.

surface and flat specimen (coated and uncoated Ti-6Al-4V alloy) by promoting a wear trail. The electric motor (Brand TEK8) is responsible for the device rotation, featuring a 12 V DC reducer, angular frequency of 50 rpm, nominal torque of 2 kgf cm, maximum torque of 6 kgf cm, reduction ratio 1:135 and mass of approximately 170 g. The sample holder, where the flat samples must be fixed for the wear tests displays a recess in the dimensions of 1.1cm x 1.1 cm. The flat samples may be locked onto the device during the tests with the aid of an 8 mm Phillips M3 screw. To ensure the transfer of charge from the top of the pen to the tip of the alumina sphere that is in contact with the substrate, a 12 mm x 21 mm x 30 mm bearing was used between the pen axis and the pen by eliminating friction between both parts. In this way, the load is transferred to the flat sample through a linear motion by producing the wear track. A threading system was chosen to facilitate the exchange of antagonists during the abrasive microwear tests in the present study. Figure 4(b) shows the equipment assembled and finished at our microwear testing facility.

3.2. Morphological Studies of the Coated and Uncoated Alloy used on Microwear Tests

Figures 5(a) and 5(b) show images acquired by SEM of the uncoated Ti-6AI-4V alloy. As shown in Figure 5(a), the sanding process promoted a roughness surface. Moreover, two distinct regions may be verified in Figure 5(b), which is related to the existence of α and β phases. SEM images of the coated Ti-6AI-4V substrate is presented in Figures 5(c, d), showing the effect of deposition on the surface of the Ti-6Al-4V alloy. It is possible to notice the deposited Nb₂O₅ coating is quite homogeneous, presenting some small regions with defects. Probably, these regions are due to the film nucleation in the initial deposition stages as already discussed in reference [4]. Further discussions have been previously published and can be found in references [4, 5]. To investigate the topography of uncoated and coated Ti-6Al-4V alloy, AFM over a scan 1.5 × 1.5 µm (base material) and 2.0 × 2.0 µm (coated material) were performed as shown in Figure 6. The presence of scratches (see Figure 6(a, b)) is related to the mechanical polishing process by using SiC sandpapers. The presence of granules in Figure 6(b) are due to the film nucleation in the initial deposition stages. SEM images were treated with the ImageJ software in search to highlight the regions of interest.



Figure 5: Surface SEM images of the Ti6-Al4-V alloy (a, b) base material and (c, d) coated with Nb₂O₅ thin film.



Figure 6: 2D and 3D morphology projection obtained by AFM mapping (a) Ti-6AI-4V alloy and (b) Ti-6AI-4V containing Nb_2O_5 thin film.

3.3. Micro-abrasive Wear Tests

Figure **7** presents a schematic assembly of the coated and uncoated specimens after the wear tests,

showing the sizes of the wear tracks as well as the used parameters. Figures 8(a, b) display the 3D representative wear tracks for the Ti-6Al-4V alloy and Ti-6Al-4V/Nb₂O₅ subjected a load of 0.5 N during 300 s. The



Figure 7: Schematic drawing of the samples appearance after the wear tests as well the used parameters.



Figure 8: (a, b) 3D representative wear tracks for the Ti-6AI-4V and Ti-6AI-4V/Nb2O5 subjected to a load of 0.5 N during 300 s and (c, d) 3D morphology of the coated and uncoated Ti-6AI-4V alloy subject to a load of 0.5 N during 600 s.

track size (1.5 mm) obtained with 3D profilometry technique confirms the design specifications and reinforces the reproducibility of the microwear tests. The peaks observed in Figure **8(a)** are probably due to changes in the orientations and size of the material's grain boundaries [14]. Figures **8(c, d)** show the wear tracks for the coated and uncoated Ti-6AI-4V alloy subjected to a load of 0.5 N during 600 s. The results

performed in both experimental test condition demonstrated that Nb_2O_5 coatings deposited on the Ti-6AI-4V surfaces through reactive sputtering technique improved the wear properties of the studied biomaterial.

Figure **9** presents a comparison of the wear volume, track depth as well as average track cross section for



Figure 9: Results obtained by 3D profilometry technique, (a) wear volume, (b) track depth as well as (c) average track cross section for the uncoated and coated Ti-6AI-4V alloy.

the coated and uncoated Ti-6Al-4V alloy obtained with 3D profilometry technique. As can be seen, the Ti-6Al-4V containing Nb_2O_5 coatings exhibited a percentage reduction on the wear volume (300 s and a load of 0.5 N) of approximately 80.1% (see Figure **9(a)**). The

cross-sections and maximum depth of the wear tracks measurements for the coated and uncoated Ti-6Al-4Valloy (see Figures 9(b, c)) corroborate the previous discussions. These findings indicated the Nb₂O₅ thin films deposited on the Ti-6Al-4V surfaces through reactive sputtering technique act as a protective barrier by improving the wear properties of the Ti-6Al-4V alloy. Although the results presented here are extremely encouraging, it is important to remember that numerous factors can influence the final wear volume. such as: characteristics of the studied material, element geometries, surface topographies, mechanical, physical. and chemical aspects, atmosphere, roughness, contact area, applied load as well as temperature of the tribological system [15-17].

The alumina spheres used during the microwear tests remained intact as verified by SEM morphological analysis and are not shown here. As the wear tracks presented an irregular surface, especially for the uncoated Ti-6Al-4V alloy, and following the criteria established by the ASTM G133 standard [6], the wear volume $(W_{v, flat})$ was determined by multiplying the average of six cross-sectional areas $(W_{a,avg})$ and the length of the wear track (S) as already ascribed by Equation 1. Here, we present a comparative study between the ASTM G133 and 3D profilometry technique only for the base material. In this sense, the average of cross-sections and standard deviation (± SD) for the uncoated Ti-6Al-4V alloy are shown in Table 1. The values obtained by the average of the sections multiplied by the length of the wear track (1.5 mm) determines de wear volume (see Table 2).

In the present work, the values obtained from the average cross-sectional areas for the Ti-6Al-4V alloy containing Nb_2O_5 thin film were not considered for the

Table 1: Average of Cross-Sections and Standard Deviation (± SD) for the Ti-6AI-4V

Load	Time	Samples	Average of cross-sections (µm ²)	(± SD)
0.5 N	300 s	Base material	21.72	6.50
	600 s	Base material	442.67	139.00

Table 2: Wear Volume Values by Comparing the 3D Profilometry Technique and ASTM G133 Standard [6] for the Ti-6AI-4V Alloy

Load	Time	Samples	3D (μm ^³)	ASTM G133 (μm ^³)	Error (%)
0.5 N	300 s	Base material	42.339	32,580	23.0
	600 s	Base material	767.353	664,000	13.5

comparison between the ASTM G133 and 3D profilometry technique. Atypical wear tracks were observed for Ti-6Al-4V alloy containing Nb₂O₅ thin films which compromised the exact determination of the cross-sectional area. These observations may be directly related to the thickness of the Nb₂O₅ coatings (~ 400 to 450 nm) produced in the present work with the reactive sputtering technique. On the other hand, as the sputtering device used for the deposition is customized, it is possible to produce coatings of different thicknesses, interfering on the wear results as presented herein. In addition, the polishing process of the samples was performed manually which can cause irregularities on the surfaces, affecting the wear results.

Another important observation made by Ayerdi et al [10], which corroborates the results of this work, is due to the difference in the wear volumes obtained by using 3D profilometry technique and the ASTM G133 standard. These authors found an error of approximately 17.8% between the two techniques while an error about 23% was verified for the base material tested during 300 s and about 13.5% for the sample's tested for 600 s in this work. Certainly, this discrepancy may be related to the morphological properties of the wear tracks. On the other hand, the 3D profilometry

technique exhibits the real wear volume, whilst the ASTM G133 [9] is directly related to the irregularities of the wear tracks.

Figure 10 exhibits the morphology of worn surfaces of coated and uncoated Ti-6Al-4V alloy obtained by SEM. Figure **10(a)** shows the wear track obtained for the Ti-6Al-4V alloy tested during 300 s. SEM analyzes show the entire wear track as well as pointed out three different regions (left side, center, and right side) (see Figures **10(b-d)**. The wear tracks for the Ti-6AI-4V allov testes during 600 s as well as Ti-6AI-4V alloy containing Nb₂O₅ thin film tested during 300 and 600 s are shown in Figures 10(e-g), respectively. Microscopic examination of the worn surfaces demonstrated a distinct behaviour among the studied materials. Considering the Ti-6Al-4V alloy, the wear tracks showed evidence of material deposits on its extremities as well as scratches. The wear properties of the Ti-6Al-4V alloy were directly affected by the incorporation of the Nb_2O_5 thin films (see Figures **10(f, g)**). In other words, the Nb₂O₅ coatings improved the wear properties of the Ti-6Al-4V alloy. Figure **11** displays the wear track obtained by using the SEM technique and the EDX maps for the Ti-6AI-4V/Nb₂O₅ specimen tested during 300 s, which allow checking the region



Figure 10: Morphologies of the worn surfaces, (a) Ti-6AI-4V alloy tested during 300 s, (b-d) different regions of the wear track (left side, center, and right side), (e) Ti-6AI-4V tested during 600 s, (f) Ti-6AI-4V/ Nb₂O₅ tested during 300 s, and (g) Ti-6AI-4V/ Nb₂O₅ tested during 600 s.



Figure 11: Wear track and EDX maps (Ti-6Al-4V/Nb₂O₅ tested during 300 s), showing the Nb₂O₅ thin film removed during the wear tests.

where the Nb_2O_5 coating was removed. In fact, the coating acted as a protective layer by increasing the mechanical properties of the Ti-6Al-4V alloy.

4. CONCLUSIONS

In the present work a low-cost ball-on-flat linear reciprocating apparatus was developed following the ASTM-133 standard recommendations. To validate the projected device, micro-abrasive wear tests were performed on uncoated and coated Ti-6AI-4V alloy with Nb₂O₅ coatings. The wear results were evaluated by using 3D profilometry technique. Our results demonstrated the Nb₂O₅ coatings deposited on the Ti-6AI-4V alloy by using reactive sputtering technique improved the wear properties of the Ti-6AI-4V alloy, reducing the wear volume about 80.1 % when compared to the base material. The 3D profilometry technique appears as a powerfull tool when compared to the ASTM G133 standard to determine the wear volume of the Ti-6Al-4V alloy. In addition, the constructed device proved to be effective and reproducible to perform microwear tests.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

DATA AVAILABILITY

Not applicable

CODE AVAILABILITY

Not applicable

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