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STUDY OF THE TRIBOLOGICAL BEHAVIOUR OF A STAINLESS-STEEL USED IN BIOMEDICAL APPLICATIONS CONDUCTED WITH LASER UNDER DIFFERENT FREQUENCIES

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Abstract. *The objective of the present work is to study the bio-tribological behaviour of the ASTM F138 austenitic stainless-steel, under different conditions of surface treatment by laser of optical fiber doped with ytterbium. Initially, the surface of four specimens individually received the incidence of ytterbium-doped optical fiber laser – pulsed, with a wavelength of 1062 nm and average power of 50 W, under an application speed of 200 mm/s and under the frequencies of 80 kHz, 188 kHz, 296 kHz and 350 kHz, related to pulse duration time values of 167 ns, 40 ns, 23 ns and 20 ns, respectively. After, the four specimens were directed to sliding tribological wear tests, under a sliding distance of 12 m and a normal force value of 1 N. To simulate the chemical action of body fluids, an aqueous solution of PBS – Phosphate-Saline Buffer was used during the experiments, dripped between "body" and "counter-body" – its chemical composition is based on a saline solution containing sodium chloride – NaCl, potassium chloride – KCl, potassium phosphate –* Na_2HPO_4 *and monopotassium phosphate –* $KHPO_4$. *The results revealed that the tribological behaviour of ASTM F138 austenitic stainless-steel was influenced by the pulse frequency of the pulsed ytterbium-doped optical fiber laser: under higher laser frequencies, lower volumes of wear were generated, because with the increase of the laser frequency application, an increase in the superficial hardness of the material was reported.*

Keywords: *Biomaterial, ASTM F138 austenitic stainless-steel, Ytterbium-doped optical fiber laser, Wear, Sliding wear, Tribological behaviour.*

1. INTRODUCTION

In today's time, there is a significant focus in the area of "*bioengineering*", developing new biomaterials and techniques that can improve their mechanical and tribological properties.

The term "*biomaterial*" is linked with biological systems including body fluids, with surgical applications and diagnostic or therapeutic vaccines, which consist of compounds of synthetic or natural origin (Pires *et al.*, 2015).

According to Park and Lakes (2007), a "*biomaterial*" is a material that is used as a replacement of parts and/or functions of the human body in a safe and reliable way.

Biomaterials are used in the manufacture of orthopedic implants and present specific conditions when under contact with the body fluids. They are in general classified into four categories, according to the compatibility that they present with adjacent tissues (Bhat, 2002; Santos, 2002):

a) *Biotolerant*: These biomaterials are used in the manufacture of orthopedic implants that remain separated from the adjacent body bone by a layer of "soft" tissue along the interface. In biotolerant synthetic polymers and metallic materials are included;
b) *Bioinert*: These biomaterials are adopted in orthopedic implants designed for applications under direct contact with bone tissue, with participation in osteogenesis having no chemical reaction between the tissue and the implant. Bioinert materials include alumina, carbon, niobium, titanium and zirconia;
c) *Bioactive*: This biomaterial is selected when there is an interaction between the orthopedic implant and bone tissue, interfering directly in osteogenesis. By chemical similarity, there is a physical connection between the mineral part of the bone tissue and the orthopedic implant, which promote osteoconduction – defined as the formation of bone in the receptor bed along the surface of the graft. In this case, the formed bone is known as "glass bone". Biomaterials like ca-phosphate, hydroxyapatite and vitro-ceramics belong to bioactive biotmaterials;
d) *Bioreabsorbable*: These biomaterials after a period of contact with human tissues, end up being degraded, solubilized or phagocyted by the body. The representatives of this class of biomaterials are tricalcium phosphate (TCP) and lactic poly-L-acid (PLLA).

Scientifically, "*biocompatibility*" can be described as the ability of a biomaterial to perform a specific function in the human body, without causing toxic or injurious effects to the biological system (Donachie, 1998). It involves a range of processes that include mechanisms of different interdependent interactions between the "*biomaterial*" and the "*body tissue*".

According to Barbucci (2002), metallic biomaterials used in the manufacture of orthopedic prostheses, osteosynthesis devices or dental implants, need to have a resistance limit of the order of 800 MPa or higher. They possess higher corrosion resistance and are biocompatible. They aim to hold the edges of fractured bones together by means of suture, ring, plate or other mechanical means via surgical intervention performed at the ends of a fractured bone. Co-Cr-Mo alloys, Co-Ni-Cr-Mo alloys, vanadium steel, pure titanium and austenitic stainless-steels falls in this category.

Studies record higher rates of use of biomaterials implants as orthopedic implants, bone prostheses and dental prostheses due to their superior mechanical properties and corrosion resistance. In Brazil, in its highest percent, orthopedic implants are manufactured mainly in metallic biomaterials, with the purpose of exchanging hard tissues, exemplifying the total replacement of hip and knee, plates and screws directed to fracture fixation, in addition to fixation devices of columns and dental implants (Wong and Bronzino, 2007).

In present context, stainless-steels are cited because they have a relatively lower cost when compared to cobalt or titanium based metals and their alloys, besides having excellent mechanical properties and chemical stability.

Austenitic stainless-steels are basically Fe-Cr-Ni alloys which are non-magnetic materials with cubic structure of centered face – CCF, and can be used in applications at low temperatures (Carbó, 2008). In addition to carbon (C), iron (Fe) and other chemical elements, austenitic stainless-steels contain a minimum amount of 10.5% chromium (Cr) – % mass, which gives them a high corrosion resistance (Carbó, 2008), by forming thin passive layer of chromium oxide (CrO_2) on its surface, which is impervious and insoluble to corrosive media. Other chemical elements such as nickel (Ni), improve the mechanical properties of austenitic stainless-steels, besides causing the transformation of the ferritic microstructure into austenitic microstructure, resulting, consequently, in major changes in its own mechanical properties.

In particular, austenitic stainless-steels are important metallic biomaterial having high chemical and mechanical stability. They contribute in increasing the half-life of prostheses implanted in the human body and avoid subsequent medical procedures to perform maintenance on them. However, ASTM F138 austenitic stainless-steel is successfully used in varied applications, in which orthopedic implants are allocated in contact with soft tissues and bones of the human skeleton. Under an estimated life-span of one decade they present a perfectly acceptable level of biological response, in addition to excellent clinical acceptance.

ASTM F138 austenitic stainless-steel corresponds to the special class of AISI 316L austenitic stainless-steel for medical bio-applications, having high applicability for such purposes (Giordani *et al.*, 2007). Also, the international standard is of greater scope to the subject which specifies the use of ASTM F138 austenitic stainless-steel as "Standard Specification for Wrought 18Cr 14Ni 2.5Mo stainless steel bar and wire for Surgical Implants" – UNSS31673 (2014).

However, no surgical implant is completely free of adverse reactions to the human body. To satisfactorily perform the function of replacing, supporting or increasing the capacity of a body structure, the implant must be compatible with the chemical and physical characteristics of the human limbs. In relation to metallic biomaterials, under their natural forms of occurrences, small amounts of fragments end up being tolerated by the human body, as iron (Fe) in red blood cells, cobalt (Co) in synthesis of vitamin B12 (Wong and Bronzino, 2007) and in the cross-links of elastin present in the aorta artery (Park and Lakes, 2007). However, in large quantities, most metals are not tolerated by the biological system of humans.

It has been observed that in biomedical applications wear is undesirable, but also inevitable and can cause numerous damage to its applications in relation to patients. The phenomenon of "*wear*" is influenced by a set of factors not inherent to the biomaterial itself but also the chemical and physical characteristics of the environment in which the mechanical component is subjected. In addition to the temporal variation of these factors type of loading, intensity, relative movement and sliding speed between surfaces, temperature and duration of the process must be taken into account.

Thus, knowing that the knowledge related to the tribological behavior of biomaterials can provide assertive decisions by professionals regarding the selection and direction of work, the objective of this work is to study the tribological behavior of ASTM F138 austenitic stainless-steel, treated superficially with fiber optic laser doped with ytterbium, under different frequencies of application and quantifying the efficiency of this type of superficial treatment for biomedical purposes.

2. MATERIALS, TRIBOMETER AND SCIENTIFIC METHODOLOGY

2.1. Materials

Four ASTM F138 austenitic stainless-steel specimen treated superficially by laser were selected for tribological tests, in addition to a fifth specimen, without surface treatment for comparative analysis.

The surface of each one of the four specimens received the incidence of ytterbium-doped fiber optic laser (Yb) – pulsed, with a wavelength of $1062^{\pm 3}$ nm and average power of 50 W, under an application speed of 200 mm/s.

Table 1 presents the frequency and pulse duration time values for the different surface treatment conditions established for the specimens.

Table 1. Frequency and pulse duration time values for the different surface conditioning conditions of the specimens.

Specimen	Frequency [kHz]	Pulse duration time [ns]
1	80	167
2	188	40
3	296	23
4	350	20

An 316L austenitic stainless-steel test ball was used as counter-body during the tribological tests.

Table 2 shows the hardness values of the specimens and the test ball.

Table 2. Hardness values of specimens and test ball.

Specimen	Hardness [HV]
as received	199
1	204
2	215
3	226
4	239
Test Ball	856

To simulate the chemical action of body fluids, an aqueous solution of PBS – Phosphate-Saline Buffer was used during the experiments. This solution is ideal for this purpose as it is based on a saline solution containing sodium chloride – NaCl, potassium chloride – KCl, potassium phosphate – Na_2HPO_4 and monopotassium phosphate – $KHPO_4$. Table 3 shows the density of each one of these chemical compounds present in the saline composition.

Table 3. Chemical composition of Phosphate-Saline Buffer (PBS).

Chemical element	Density [g/l]
NaCl	8
KCl	0.2
Na_2HPO_4	1.15
$KHPO_4$	0.2

2.2. Tribometer

Figure 1 shows the "*ball-cratering*" tribometer used in this work. Having "*fixed-ball*" mechanical configuration, the test shaft was divided into two distinct parts, called "*motor test shaft*" and "*moving test shaft*" (Figure 1a). In turn, each of these parts has a face with concave radius of R_{sphere} = 12.7 mm (R_{sphere} = ½"), thus enabling the accommodation of a test sphere of diameter D = 25.4 mm (D = 1" – *standard size*). For the application of the normal force, a "*dead-weight*" system was adopted (Figure 1b).

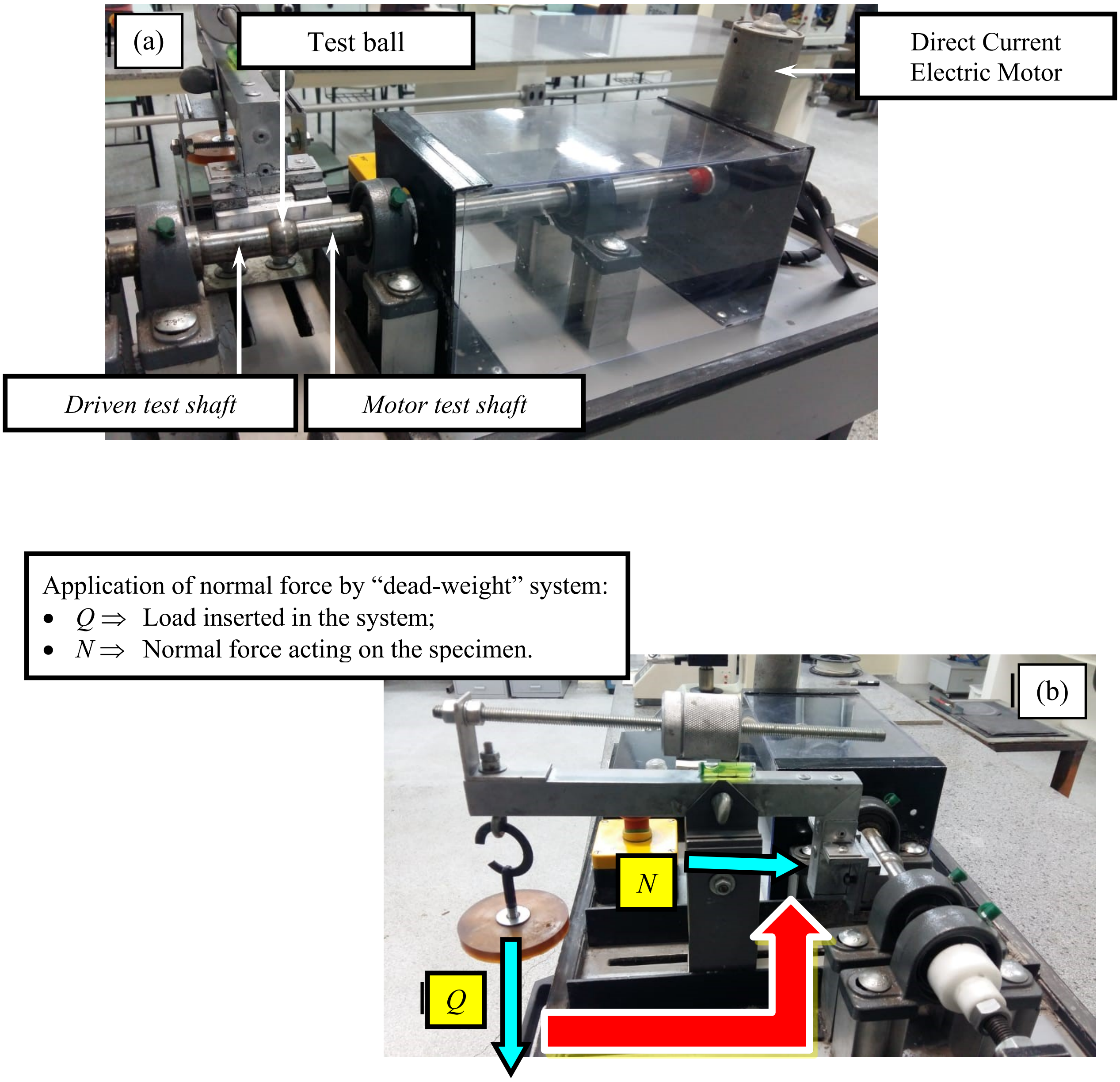

Figure 1. "*Ball-cratering*" tribometer of "ball-fixed" mechanical configuration: (a) "Motor test shaft", "test ball" and "driven test shaft" mounted on the tribometer; (b) application of normal force by "dead-weight" system.

2.3. Scientific Methodology

A normal force value of 1 N was defined for tribological tests, along with a rotation of the test sphere of 50 rpm – resulting in a tangential velocity of 0.066 m/s. For a sliding distance set at 12 m, the required test time was calculated at 3 min. Table 4 summarizes the test conditions established for this study.

Table 4. Test conditions established for the tribological tests.

Test parameter	Value
Normal force – *N*	1 N
Rotation of the test ball – *n*	50 rpm
Tangential ball speed – *v*	0.066 m/s
Test time – *t*	3 min
Sliding distance – *S*	12 m

All experiments were conducted without interruption and the Phosphate-Saline Buffer (PBS) chemical solution was continuously inserted between the specimen and the test ball during the wear tests, at a frequency of 1 drop every 2 s.

At the end of the wear tests, the diameters of the wear crater (*d*) wear were measured by optical microscopy. Then, the wear volumes (*V*) were calculated by Equation 1, in which *R* is the radius of the test sphere.

$$V \approx \frac{\pi d^4}{64R} \qquad \text{for } d << R \tag{1}$$

After the analysis by optical microscopy, images were detailed by scanning electron microscopy, in order to verify the tribological conditions of the surfaces of the wear craters.

3. RESULTS AND DISCUSSION

3.1. Overview

One of the great challenges directly involved in performing orthopedic surgeries is the development of implants whose purpose is the fixation of fractures or the replacement of parts of the human skeleton. To this end, these mechanical devices must be manufactured of materials that reconcile significant mechanical-tribological performance and convenient compatibility with the organism of humans (Chohfi *et al.*, 1997).

Surface treatment by laser of fiber optic doped with ytterbium was able to produce microstructural transformations that promoted the increase of the wear resistance of the ASTM F138 austenitic stainless-steel, due to the surface heating of the material by the absorption of the incident radiation in it. In this type of treatment, the increase in temperature is extremely fast up to the austenitization range, due to the concentration of energy in thin layers; for the turn, cooling, depending on the underlying cold layers, will also occur relatively quickly, producing the effect of quenching heat treatment in the heat affected zone, with-out the need for the use of a cooling liquid (Abdalla *et al.*, 2006; Assumpção and d'Oliveira, 2001).

3.2. Analysis of the surfaces of the wear craters

Worn surfaces of wear craters were studied and it was observed that wear marks produced were due to relative sliding motion between the test sphere and the specimen, resulting in progressive loss of material due to the continuous formation and destruction of the roughness peaks. This type of contact condition is one of the physical factors that limit the performance and useful life of a biomechanical component. As majorly biomechanical components are used in implants performing under dynamic loads, generating friction and, consequently, compromising the mechanical and tribological integrity leading to its premature failure.

Figure 2 presents images of wear craters surfaces generated during the wear tests.

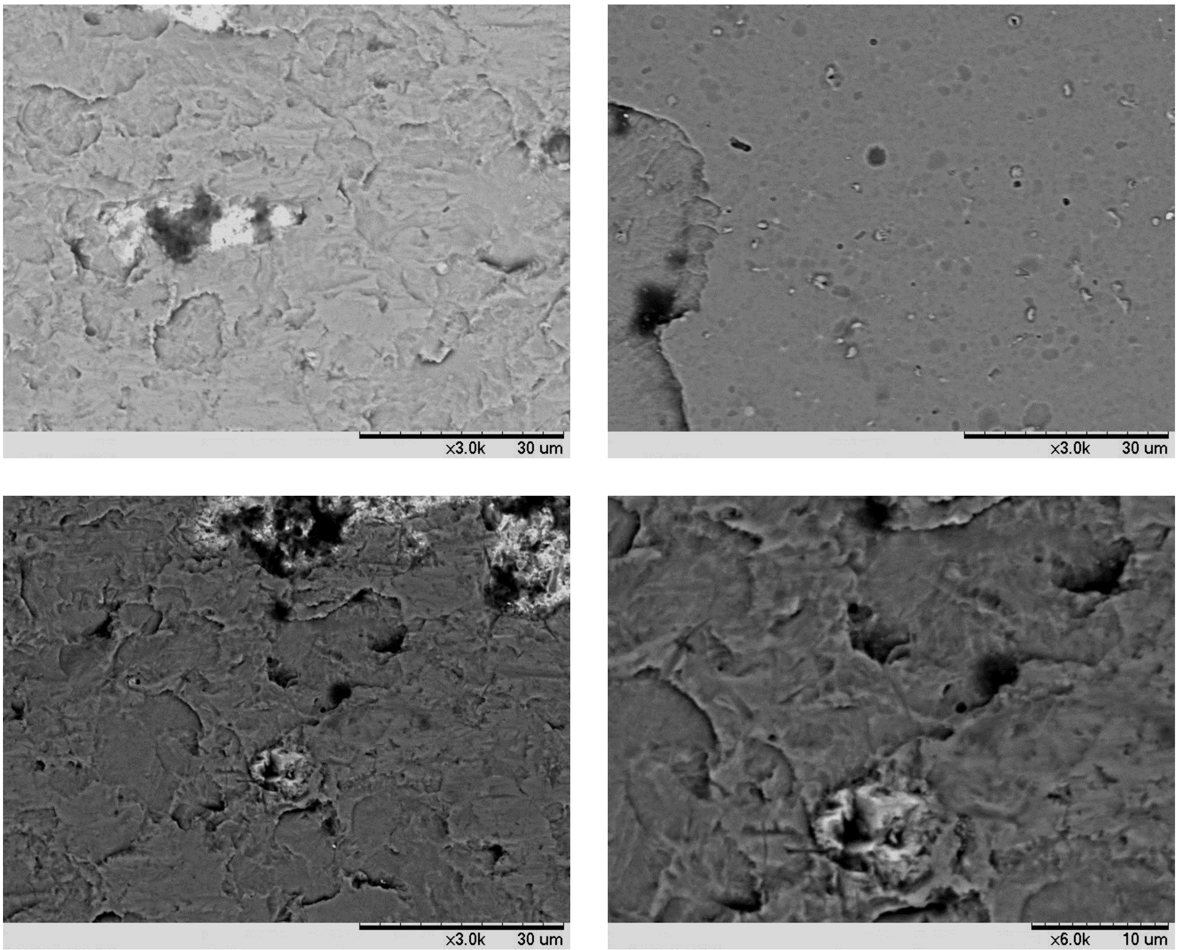

Figure 2. Images of wear craters surfaces generated during the wear tests.

3.3. Wear resistance analysis

The surface treatment with fiber optic laser doped with ytterbium promoted the surface hardening of ASTM F138 austenitic stainless-steel, due to the heating of the surface of the specimens below the melting temperature, which caused a phase transformation in the solid state without altering the microstructure of the substrate.

Figure 3 presents the graph of the results obtained for the analysis of tribological behavior in relation to the wear volume (V) of the specimens, as a function of the frequency of surface treatment of the optical fiber laser doped with ytterbium – $V = f(f)$.

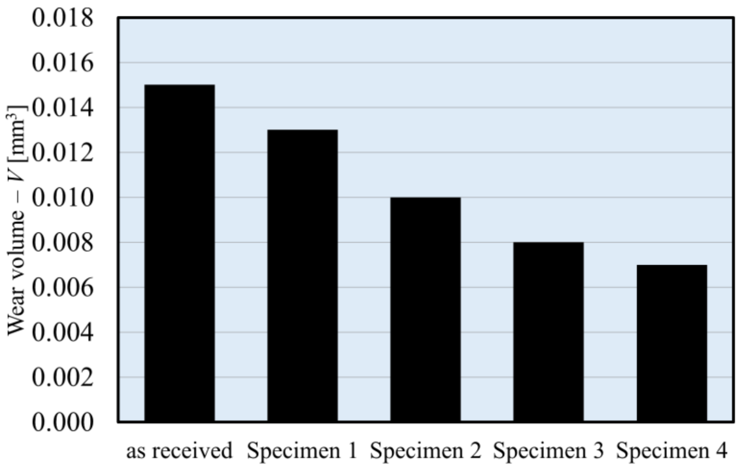

Figure 3. Wear volume (V) as a function of the frequency of surface treatment of the optical fiber laser doped with ytterbium – Maximum standard-deviation of V: $SD_V = 0.001$ mm^3.

Increase in pulse frequency (*f*) of the fiber optic laser doped with ytterbium caused an increase in the surface hardness of the material analyzed and, consequently, an increase in its wear resistance, characterized by reduction of the wear volume (*V*).

4. CONCLUSIONS

Surface treatment with fiber optic laser doped with ytterbium generated an increase in the surface hardness of ASTM F138 austenitic stainless-steel and, consequently, an increase in its wear resistance.

With increase in laser frequency application, an increase in the superficial hardness of the material was observed with decrease in wear volume.

Surface treatment by optical fiber laser doped with ytterbium showed satisfactory results regarding increase in wear resistance of the biomaterial. Therefore, it can be concluded that it is an applicable procedure for the conditioning of biomaterials that aim to prolong its useful life.

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