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Characterization of physical properties of bismuth-titanium coatings on 316L stainless steel

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Abstract. The 316 L steel is a type of stainless steel widely used in the medical industry, which in recent years has been studied for different uses in society. Being an engineering material, it is imperative to know its performance based on its physical and mechanical properties that allow identifying the response of this steel in addition to thin films as coatings. Bismuth and titanium have been recently used to improve the properties of 316 L steel, so they were used in this study. The sol-gel technique was used as the film forming method. The response of physical and mechanical properties was evaluated from the analysis of microhardness and coefficient of friction reported for the different types of steel-coating systems. Higher microhardness values were found for films with higher proportion of titanium. The coefficient of friction values is influenced by the system used, with higher values obtained for samples with a single coating layer.

1. Introduction

The use of coatings is an alternative to extend the useful life of materials and provide additional protection without affecting their basic properties, acting as an anti-wear barrier by reducing the possibility of contact or friction between the material and the surrounding environment. Coatings also allow to promote adhesion, or thin inert coatings on wear resistant layers to reduce tool corrosion. Some studies have shown that the use of several layers of coatings improves the hardness, toughness, and adhesion values of materials [1].

We have chosen to work on AISI 316 L stainless steel because it has been widely used in different industrial contexts, being an alternative to titanium due to its low cost. However, it has some disadvantages in terms of problems such as corrosion when subjected to contact with saline substances. Likewise, this type of steel has demonstrated low mechanical strength and poor anti-friction properties, which has hindered some engineering applications with higher requirements [2].

The sol gel method has been widely used to obtain thin films or coatings, from the hydrolysis and condensation of precursors or alkoxide chemical components, being a film production technique, with efficient adhesion to the substrate [3]. The sol-gel method is a material synthesis process of great interest nowadays; this procedure consists of three main parts: (a) sol preparation, (b) sol gelation, and (c) solvent removal [4].

Bismuth (Bi) and titanium (Ti) have been elements for which many results of combined use have been reported for the improvement of properties of steels, from the production of coatings or thin films;

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finding for example that the thin films deposited on steel of these elements on 316 L steel present a low roughness and a homogeneous surface in general [3]. Likewise, some favorable results have been found in terms of biocompatibility [5,6].

Therefore, the objective of this article is to analyze the behavior of the physical and mechanical properties of 316L steel in the presence of Bi-Ti ceramic coatings, allowing to expand the scientific knowledge oriented to explore new uses of this material in society.

2. Methodology and materials

The precursors used for the conformation of the coatings in the present article were titanium (IV) butoxide ($Ti(OBu)_4$) and bismuth (III) nitrate pentahydrate ($Bi(NO_3)_3 \cdot 5H_2O$). To evaluate and compare the effectiveness of the molar ratio of each of the precursors, three types of combinations were worked with, as shown in Table 1.

Table 1. Experimental design and characteristics of the samples used.

Concentration	Spin speed (rpm)	Number of layers applied
Bi-Ti (70-30)	3000, 4000, and 5000	Monolayer, bilayer
Bi-Ti (80-20)	3000, 4000, and 5000	Monolayer, bilayer
Bi-Ti (90-10)	3000, 4000, and 5000	Monolayer, bilayer

The sintering of the films was carried out based on the technique used in [7], to guarantee a controlled elimination of organic components and the stability of the substances obtained to produce the coatings. To measure the hardness of the substrate-coating systems, nanoindentation tests were performed with the INNOVATEST® Impressions XT device for Vickers hardness at 0.3 Kgf.

3. Results and discussion

The analysis of the physical and mechanical properties considered in the research is presented in two sections: micro-hardness reported in each of the films deposited on the steel and coefficient of friction of the different combinations studied.

3.1. Microhardness

For the microhardness test, 4 measurements per sample were performed and a load of 0.3 Kgf was used for 10 seconds (s). Table 2 shows the average Vickers hardness (HV) values for all the proposed systems. Slightly higher hardness values are observed for the 70-30 system and 80-20 system, which can be attributed to the higher proportion of Ti present, since it has been reported that titanium oxides contribute higher hardness to thin films [8]. All the films present a hardness higher than that of the uncoated 316 L steel, it is noteworthy the sample 80-20, monolayer, 3000 rpm which reports the highest hardness value of 204.12 HV obtaining an increase of 13% with respect to the steel.

The fact that lower hardness values are obtained for some bilayer samples may be due to the growth of oxides in the films. As can be seen in Table 1, there is no strong influence between the hardness and the change of the centrifugation speeds nor from the number of layers; likewise, there are no significant variations in the hardness values of all the samples, which can be attributed to the fact that the grain size within the films is similar, as suggested by Bull, *et al.* [1]. Likewise, according to Li, *et al.* [9], decreasing the grain size increases the hardness.

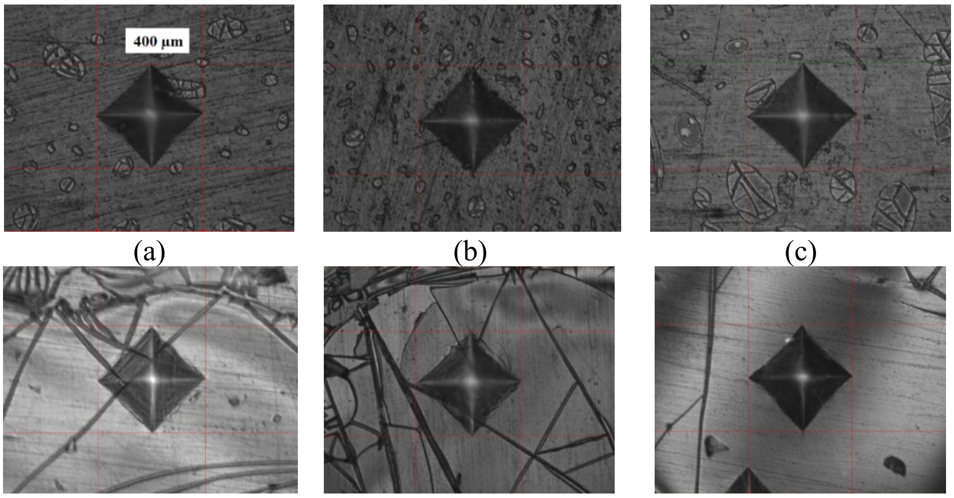
Table 2. Microhardness values of the synthesized coatings.

Spin speed	Bi-Ti (70-30)		Bi-Ti (80-20)		Bi-Ti (90-10)	
	Monolayer	Bilayer	Monolayer	Bilayer	Monolayer	Bilayer
3000 rpm	183.78±4.9	177.69±3.2	192.24±15.6	194.42±4.1	183.32±6.7	185.10±6.0
4000 rpm	181.48±1.3	190.65±14.2	192.32±4.6	185.32±5.9	183.05±7.4	180.46±7.3
5000 rpm	187.76±13.9	182.28±13.8	187.16±5.3	184.03±5.4	182.69±7.1	184.43±5.5

Figure 1 and Figure 2 show images of the indentation traces on the 70-30 film and 80-20 film at the three proposed speeds, monolayer, and bilayer, respectively, which are produced by the diamond tip indenter when applying the Vickers hardness test.

The films present stacking, which can be evidenced by the presence of bright halos at the edges of the indentation [10], this can generate radial stresses and cracks in the form of circumference which is due to the fact that the film is forced to follow the plastic flow of the substrate, in this case steel [11], this type of cracks are quite noticeable in the films 70-30, bilayer 3000 rpm and 4000 rpm and 80-20, bilayer, 4000 rpm, the amount of stacking depends on the hardening capacity of the film.

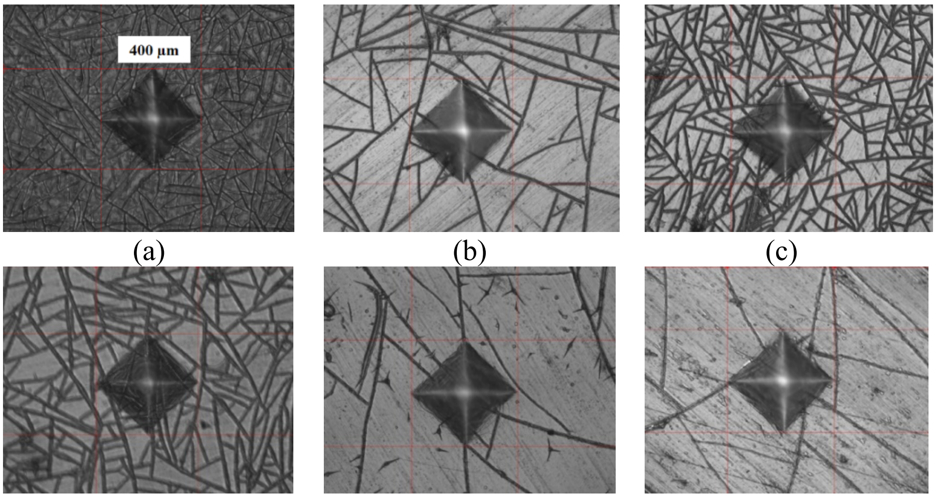
Similarly, ring or nest type cracks are observed within the indentation trace, which are propagated by the action of tensile stresses, these are quite evident in the samples, bilayer, 3000 rpm at 70-30 and 80-20 Bi-Ti; for the three sample combinations, quite heterogeneous surfaces are observed. Samples 90-10, as shown in Figure 3, show no cracking and a formation of bismuth granules.



(a) (b) (c)

(d) (e) (f)

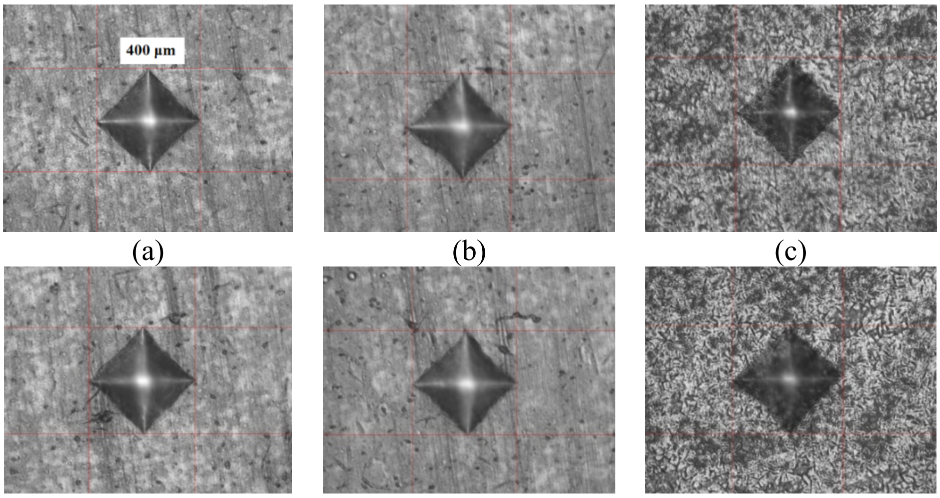
Figure 1. Vickers hardness (HV 0.3), Bi-Ti (70-30) films; (a) monolayer - 3000 rpm; (b) monolayer - 4000 rpm; (c) monolayer - 5000 rpm; (d) bilayer - 3000 rpm; (e) bilayer - 4000 rpm; (f) bilayer - 5000 rpm.



(a) (b) (c)

(d) (e) (f)

Figure 2. Vickers hardness (HV 0.3) Bi-Ti (80-20) films; (a) monolayer - 3000 rpm; (b) monolayer - 4000 rpm; (c) monolayer - 5000 rpm; (d) bilayer - 3000 rpm; (e) bilayer - 4000 rpm; (f) bilayer - 5000 rpm.



(a) (b) (c)

(d) (e) (f)

Figure 3. Vickers hardness (HV 0.3) of Bi-Ti (90-10) films; (a) monolayer - 3000 rpm; (b) monolayer - 4000 rpm; (c) monolayer - 5000 rpm; (d) bilayer - 3000 rpm; (e) bilayer - 4000 rpm; (f) bi-layer - 5000 rpm.

3.2. Coefficient of friction

Figure 4 shows the coefficient of friction (COF) curves for the films synthesized at 70-30 (Bi-Ti) monolayer and bilayer at centrifugation speeds of 3000 rpm and 5000 rpm. For the samples at 4000 rpm, bilayer, COF values close to 0.09 are reported, which remains constant until the end of the test. In the case of the bilayer samples at 3000 rpm, COF values between 0.06 and 0.07 are evidenced during the 5 and 45 seconds of the test and for the last 15 seconds, the COF presents a slight significant decrease up to 0.065, which can be attributed to adhesive wear and film detachment. It is noteworthy that the COF values start from very low values, which could be due to a low surface roughness value of the sample that delays the formation of residues, delaying the abrasion damage [12].

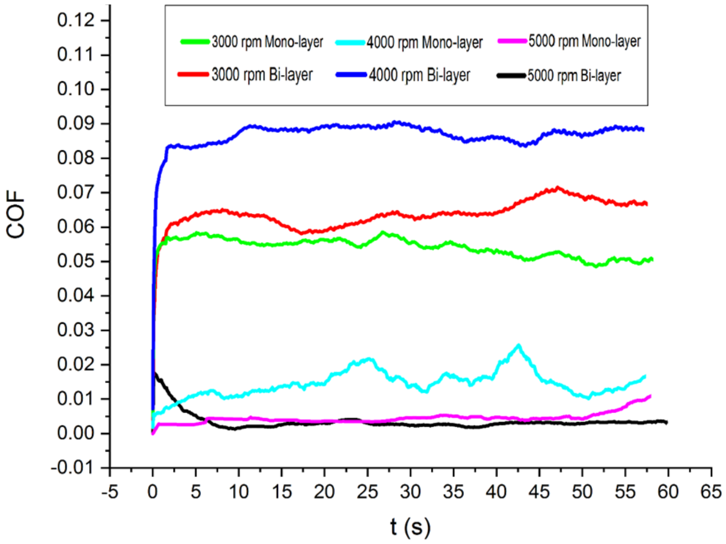


Figure 4. Coefficient of friction concentration Bi-Ti (70-30).

The COF values for the Bi-Ti (80-20) mono and bilayer films are presented in Figure 5. The bilayer films report the highest COF values. COF values of 0.16 to 0.18 are considered for the coating at 5000 rpm bilayer and 0.08 to 0.10 for the film at 3000 rpm bilayer. It is observed that the centrifugation speed is not a statistically representative variable, as is the number of layers deposited.

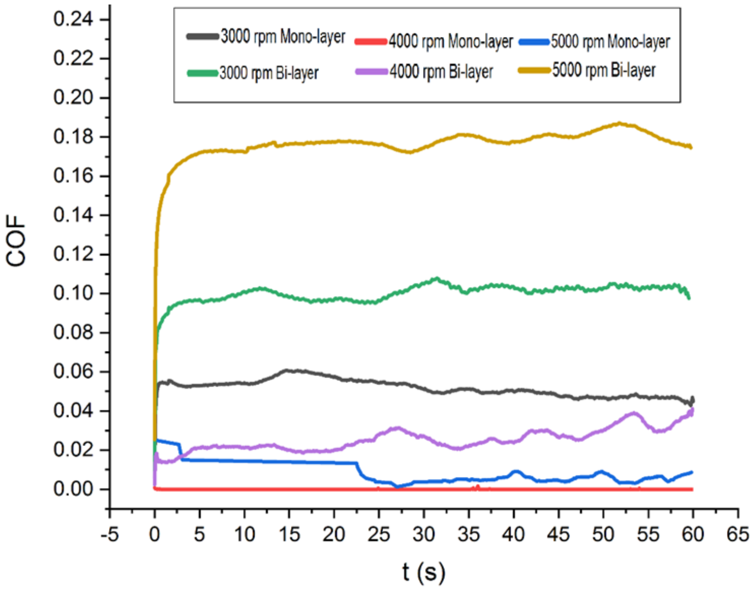


Figure 5. Coefficient of friction concentration Bi-Ti (80-20).

Figure 6 shows the coefficient of friction curves for the films synthesized at 90-10 (Bi-Ti) monolayer and bilayer at selected centrifugation speeds. For the monolayer sample at 4000 rpm, there is a COF between 0.23 and 0.28 in the first 15 seconds of the test and for the last 35 seconds, the graph shows a COF value ranging between 0.27 and 0.26. As for the monolayer at 5000 rpm, a very stable and low COF value between 0.0 and 0.02 is observed during the whole test. With respect to the bilayers, fluctuations are observed throughout the test, as evidenced by the abrupt changes in the COF graphs as a function of time.

The values are influenced by the number of films deposited, obtaining higher values for monolayer samples, similarly large variations in friction during the test and can be attributed to differences in film structure or surface irregularities due to roughness [13,14], low COF values are recorded for bilayer samples. No strong influence is evident in terms of centrifugation speed and COF values.

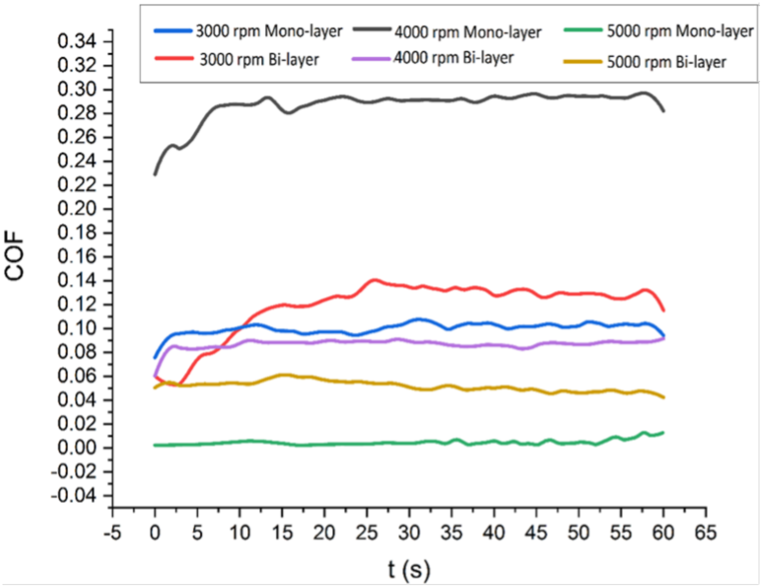


Figure 6. Coefficient of friction concentration at Bi-Ti (90-10).

4. Conclusions

Bismuth-titanium films were synthesized on 316 L steel substrates with molar concentrations (bismuth-titanium) of 70%-30%; 80%-20% and 90%-10%, respectively, by spin coating technique, varying the speeds from 3000 rpm to 5000 rpm with monolayer and bilayer coating system. This allowed us to propose an experimental design that would contribute to the comparison of factors and response variables in the efficiency of some mechanical properties.

Regarding the mechanical characterization, higher microhardness values were reported for the films with higher proportion of titanium; however, all the samples showed a hardness higher than that of uncoated 316 L steel. On the other hand, lower wear values were obtained for the samples with lower Bismuth content; however, all films evidenced to offer effective protection to the steel as they showed lower wear rates than the substrate.

The values of the coefficient of friction were influenced by the system used, obtaining higher values for the monolayer samples. It should be noted that the COF values start with very low values, which could be due to low surface roughness. The stiffest films are obtained with the samples with higher titanium content.

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