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Evaluation of micro-abrasion-corrosion on $\text{SiO}_2\text{-TiO}_2\text{-ZrO}_2$ coatings synthesized by the sol-gel method

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Abstract. The medical science and the engineering, work to improve the materials used in the manufacture of joint implants, since they have a direct impact on the quality of people life. The surgical interventions are increasing worldwide with a high probability of a second or even a third intervention. Around these circumstances, it was evaluated the behaviour against micro-abrasion-corrosion phenomena on SiO_2 , TiO_2 , ZrO_2 coatings, synthesized by the sol-gel method with concentration of the Si/Ti/Zr precursors: 10/70/20 and 10/20/70. The coatings were deposited on AISI 316 LVM stainless steel substrates. The morphological characterization of the wear was made by AFM techniques. It was observed that the coatings with higher levels of titanium have a good response to the phenomena of microabrasion-corrosion.

1. Introduction

The main characteristic of stainless steel is its high resistance to corrosion, but when getting in contact with Cl^- and Br^- they undergo of pitting. The sol-gel is one of the methods of synthesis of materials that presents great interest at the present time; by means of this method it is possible to obtain ceramic coatings that act as an anticorrosive barrier. Research projects carried out on this topic have indicated that sol-gel method has allowed applying binary systems ($\text{SiO}_2\text{-TiO}_2$ and $\text{SiO}_2\text{-ZrO}_2$) on stainless steel substrates AISI 304 and has allowed to diminish the attack of corrosive agents [1].

The sol-gel method consists of three main parts: a) the preparation of the sol, b) gelation of the same, and c) removing the solvents [2-5]. This technology has been widely applied in different fields of materials science, [6-11].

In the present study, system $\text{SiO}_2\text{-TiO}_2\text{-ZrO}_2$ was synthesized with the sol-gel method at room temperature. These stable soles were formed in concentrations Si/Ti/Zr: 10/70/20 and Si/Ti/Zr: 10/20/70 using as precursors tetraethyl orthosilicate $\text{Si}(\text{OC}_2\text{H}_5)_4$, titanium tetrabutoxide $\text{Ti}(\text{O}i\text{Bu})_4$ and zirconium tetrabutoxide $\text{Zr}(\text{OC}_3\text{H}_7)_4$. The morphological characterization of the wear was made by Atomic Force Microscopy (AFM) technique. It was observed that the coatings with higher levels of titanium have a good response to the phenomena of microabrasion-corrosion.

2. Experimental procedure

The process developed for the formation of the sols was as follows [1,12]. The substrates used for depositing the multi-component system, were steel sheets AISI 316LVM with dimensions of 3.5cmx2.5cmx0.2cm, which were polished to metallographic brightness. The next step was to form films by dip-coating, at a velocity of 13.2cm/min. The films sintering were carried out at heating rate of



1°C/min. The initial temperature was 20°C to 300°C and stabilized at this temperature for one hour. Heating was restarted to 400°C and equilibrated for 30 minutes.

The micro-abrasion-corrosion tests were carried out using an equipment which has a sample holder attached to a lever arm, which rotates on its pivot that controls the load on the specimen upon contact with the ball by the movement of a dead weight and a counter weight. The sphere is fixed between two coaxial shafts supported on bearings, when one of them is driven by a DC motor with encoder to ensure the speed and number of revolutions of test (see Figure 1). In order to observe the micro-abrasive process was provided abrasive particles to the system by a peristaltic pump controlled manually, these particles were suspended in Ringer's solution with a magnetic stirrer. For the study of the corrosive phenomena it was adapted to the equipment a potentiostat that contains an electrochemical cell.

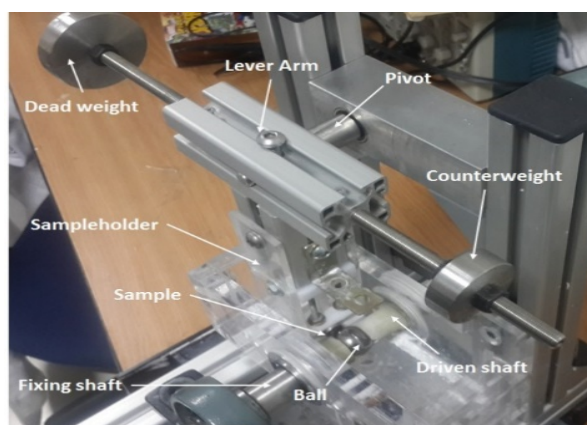


Figure 1. Micro-abrasion equipment.

Two types of tests were performed, the first tests were performed with only micro-abrasive wear, due to it was not used the electrochemical cell. Followed by micro-abrasion-corrosion tests where the wear tests were repeated with the same parameters but using the electrochemical cell, performing the electrochemical impedance spectroscopy (EIS) technique. The used parameters in tests are in the Table 1.

Table 1. Tests parameters.

| Specifications micro-abrasion test (wear only) | |
|--|---|
| Sample material | 316 LVM steel, SiO ₂ -TiO ₂ - ZrO ₂ 10/70/20 and 10/20/70 coatings |
| Ball material | AISI 52100 steel (25mm of diameter) |
| Particles size | Alumina 0.3μm |
| Solution | Ringer's solution |
| Abrasive concentration | 0.02% |
| Rotation speed | 40rpm |
| Load | 2N |
| Number of revolutions | 100 revolutions |
| Sliding distance | 15.7m |
| Slurry feed rate | 30ml/min |
| Specifications micro-abrasion-corrosion test (wear with corrosion) | |
| Reference electrode | Ag/AgCl |
| Auxiliary electrode | Platinum wire |
| Working electrode | Samples (316 LVM steel, SiO ₂ -TiO ₂ - ZrO ₂ 10/70/20 and 10/20/70 coatings) |
| EIS | Frequency: 10kHz to 0.1Hz |

3. Results and discussion

3.1. Wear rate estimation

For the wear estimations a mathematical model was used, this model relates the dimensions of the wear track with a loss volume rate and a wear constant. Therefore, after each test were taken parallel and perpendicular measurements of the generated wear scar according to the rotation of the ball and then determine their average, see Figure 2.

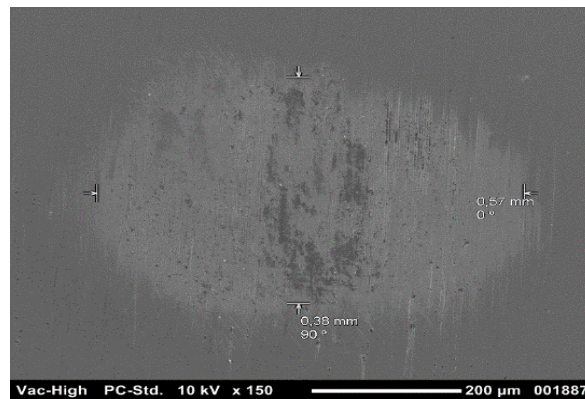


Figure 2. Measurement on wear scar of the 10/70/20 coating using SEM.

With the average lengths, the parameters of normal load (2N) and the sliding distance (15.7m) were used to calculate the volumes and constants of wear for the different performed tests as is shown in the Table 2; where is appreciated a lower wear volume for the substrate, followed by the 10/20/70 coating. The higher wear volume was exhibited by the 10/70/20 coating, however when is subjected to the micro-abrasion-corrosion presents a better behaviour in contrast with the micro-abrasion tests and the behaviour of the substrate and the 10/20/70 coating.

Table 2. Volumes and constants of wear.

| Samples | Wear volume (mm ³) | Wear constant (m ² /N) |
|---------------------------------------|--------------------------------|-----------------------------------|
| (316 L) Micro-abrasion | 1.70x10 ⁻⁰⁵ | 5.41x10 ⁻¹⁶ |
| (316 L) Micro-abrasion-corrosion EIS | 4.54x10 ⁻⁰⁵ | 1.45x10 ⁻¹⁵ |
| 10/70/20 Micro-abrasion | 1.93x10 ⁻⁰⁴ | 6.15x10 ⁻¹⁵ |
| 10/70/20 Micro-abrasion-corrosion EIS | 1.00x10 ⁻⁰⁴ | 3.18x10 ⁻¹⁵ |
| 10/20/70 Micro-abrasion | 3.48x10 ⁻⁰⁵ | 1.11x10 ⁻¹⁵ |
| 10/20/70 Micro-abrasion-corrosion EIS | 6.41x10 ⁻⁰⁵ | 2.04x10 ⁻¹⁵ |

3.2. Morphological study

In the Figure 3 is observed the surfaces before and after the micro-abrasion-corrosion tests using the AFM technique, where the 316 LVM substrate and the 10/20/70 coating presents similar rugosity in comparison with the 10/70/20 coating that exhibit a lower value due to its high Ti content (see Table 3).

Table 3. Rugosity of the substrate and the coatings before and after micro-abrasion-corrosion

| Sample | Rugosity Ra (nm) | |
|----------|------------------|------------|
| | Before test | After test |
| 316 LVM | 9.5712 | 169.81 |
| 10/70/20 | 9.0644 | 67.746 |
| 10/20/70 | 8.9521 | 135.39 |

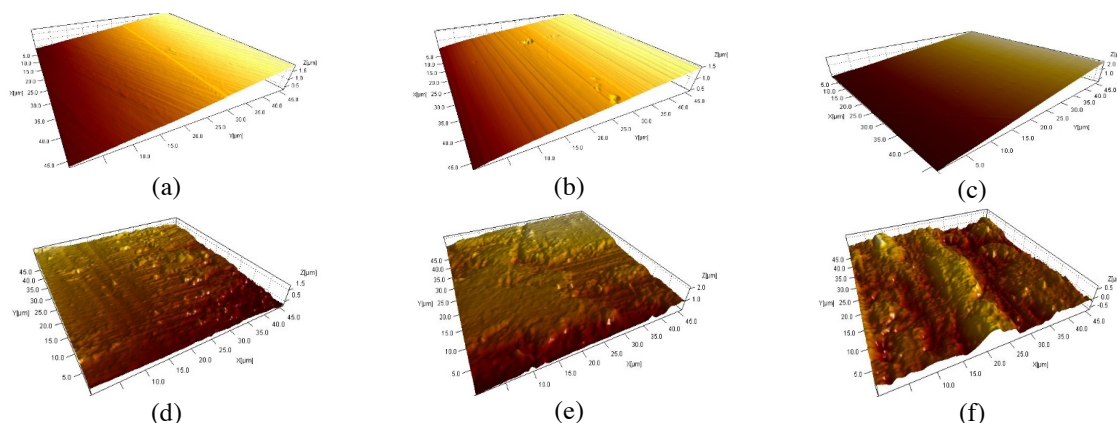


Figure 3. AFM micrographs of the surfaces before the tests (a) 316 LVM substrate, (b) 10/70/20 coating, (c) 10/20/70 coating; and the worn surfaces (d) 316 LVM substrate, (e) 10/70/20 coating, (f) 10/20/70 coating.

4. Conclusion

Despite of the higher values of wear volume exhibited by the 10/70/20 coating, it had a good performance due to the reduction of the wear volume when is subject to corrosive phenomena in contrast with the presented by substrate and the 10/20/70 coating which increase. Furthermore, the 10/70/20 coating presents the lowest rugosity at the micro-abrasion-corrosion mechanism and a similar morphology with the 10/20/70, where it was observed small grooves characteristic of the micro-abrasive wear.

Acknowledgments

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