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Adhesion and wear study on 316L steel coated with titanium nitride by PVD

Estudio de adhesión y desgaste en acero 316L recubierto con nitruro de titanio por PVD

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Abstract

The AISI 316L steel is a material used to manufacture prostheses and medical instruments due to its high corrosion resistance. However, its wear resistance is low, but it can be improved by synthesizing thin films of titanium nitride (TiN) on the surface. This compound is a hard material that improves its tribological and corrosive properties, in addition to presenting good adhesion to the substrate. In this work, a Ti/TiN thin film was obtained using the Physical Vapor Deposition (PVD) technique. This coating was characterized by scanning electron microscopy, energy dispersion spectroscopy (EDS), and light microscopy; additionally, tribological tests under wet conditions and adhesion tests were conducted. The coating showed good adhesion to the substrate according to the VDI standard. In tribological tests, a low coefficient of friction vs. a polyethylene pin in a wet environment (bovine serum) was obtained. The EDS showed a high concentration of Ti and N on the substrate even after wear tests.

TiN, Steel 316L, Tribology, PVD

Resumen

El acero AISI 316L es un material empleado para la fabricación de prótesis e instrumental médico por su alta resistencia a la corrosión. Pero debido a sus propiedades tribológicas son deficientes, puede ser mejorado superficialmente sintetizando películas delgadas de nitruro de titanio (TiN). Este compuesto es un material duro que permite mejorar sus propiedades tribológicas y corrosivas, y presenta buena adherencia al sustrato. En este trabajo se obtuvo una película delgada de Ti/TiN mediante la técnica de Deposición Física fase Vapor (PVD). Este recubrimiento se caracterizó mediante microscopia electrónica de barrido, análisis de dispersión de energía (EDS), microscopia óptica; además, se realizaron pruebas tribológicas en vía húmeda y pruebas de adherencia. El recubrimiento mostró buena adherencia de acuerdo con la norma VDI. En cuanto a las pruebas tribológicas, se observó un bajo coeficiente de fricción vs un pin de polietileno en vía húmedo (suero de bovino), el EDS mostró una alta concentración de Ti y N sobre el sustrato, aun después de las pruebas de desgaste.

TiN, Acero 316L, Tribología, PVD

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Introduction

The physical vapor deposition (PVD) process is a vaporization coating technique that transfers material at the atomic level, the element of interest to be deposited is evaporated in the vapor phase by physical methods. The process occurs in a controlled atmosphere that uses inert gas to transport; evaporated material settles on the substrate, forming a thin film as it condenses [1].

The PVD process fabricates thin films [2] and nanomaterials [3]. The process improves the properties of the substrate, which can be made of different types of inorganic materials and some organic materials; it is a process that does not generate pollutants, unlike others. Smooth surfaces can be manufactured at low temperatures, obtaining excellent mechanical, tribological, and adhesion properties [3].

On the other hand, it is known that metallic components fail by wear and corrosion, the latter due to the environment in which they are found; these factors decrease their lifetime and functionality. This problem can be reduced considerably by adding elements on the surface of metallic materials, such as titanium nitrides [4].

Titanium nitride has diverse properties according to its composition and crystalline structure, which depend on the growth characteristics controlled by deposition parameters [5].

Methodology

AISI 316L steel was used, and the samples were prepared with SiC sandpaper and diamond paste to obtain a mirror finish. The physical vapor deposition (PVD) process began with an ionic cleaning with argon for 3 minutes; subsequently, an adhesion layer of Ti was generated for 5 minutes. Next, a TiN coating was synthesized using a 99.999% pure titanium target and a constant flow of nitrogen in a controlled atmosphere. The reactive process lasted 30 minutes.

Results

Thin Ti/TiN films were deposited on a 316L steel substrate using the PVD technique (Fig. 1); the samples were characterized by scanning electron microscopy, optical microscopy, and tribological and adhesion tests.

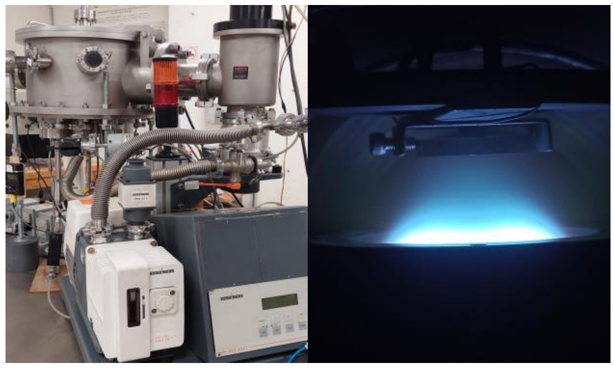

Figure 1 Equipment and PVD process on 316L steel [6]

Fig. 2 presents the EDS analysis of the substrate with and without coating. It can be observed that the presence of the alloying elements of 316L steel and Ti and N is attributed to the coating.

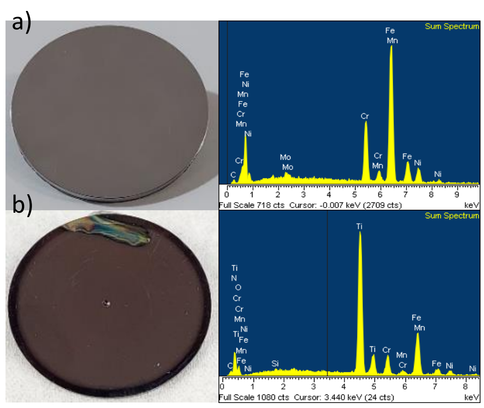

Figure 2 Energy dispersion analysis of the substrate before (a) and after coating (Ti/TiN) (b)

The adhesion test of the Ti/TiN coating was evaluated using the VDI 3198 standard (Fig. 3) [7], a Rockwell C indentation with a load of 150 kg (1471 N) was performed, the indentation footprint was compared with a reference failure map. The results indicated that the Ti/TiN coating presented an acceptable adhesion since it does not present critical fractures at the edges of the indentation (Fig. 4).

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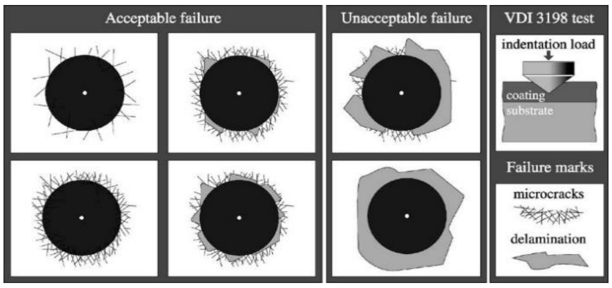

Figure 3 VDI 3198 Indentation Test Principle [7]

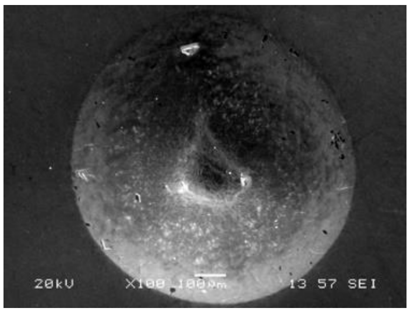

Figure 4 Ti/TiN coating adhesion test

The wear tests were conducted under wet conditions (bovine serum) against a polyethylene pin, with a total distance of 200 m and a load of 5 N. This test was performed on a coated and uncoated 316L steel substrate. The coefficient of friction obtained during the wear test showed a higher value at the beginning, then decreasing over time and finally reaching steady state. The lowest coefficient of friction was obtained on the Ti/TiN coated substrate. In addition, the uncoated substrate shows deeper wear marks compared to the coated sample (Fig. 5).

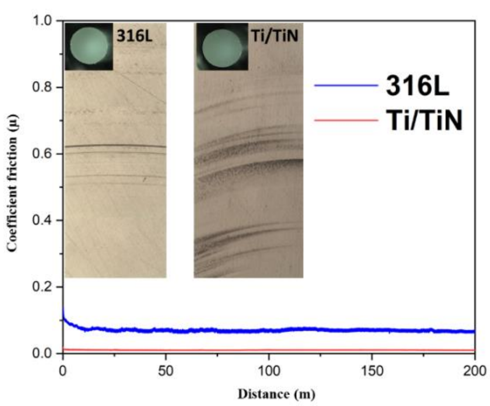

Figure 5 Wear test results.

Fig. 6 shows the EDS results of uncoated sample, it shows the concentration of Fe, Ni and Cr.

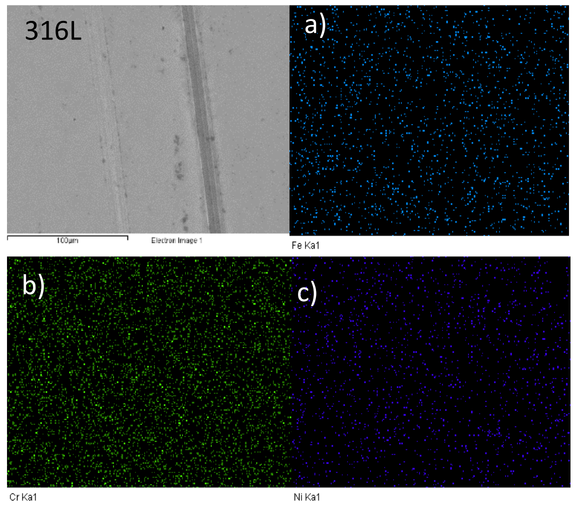

Figure 6 Analysis of elements of the uncoated 316L sample a) iron, b) chromium, c) nickel

Fig. 7 shows the concentration of Ti and N in the coated substrate after the wear test, confirming the presence of titanium nitride.

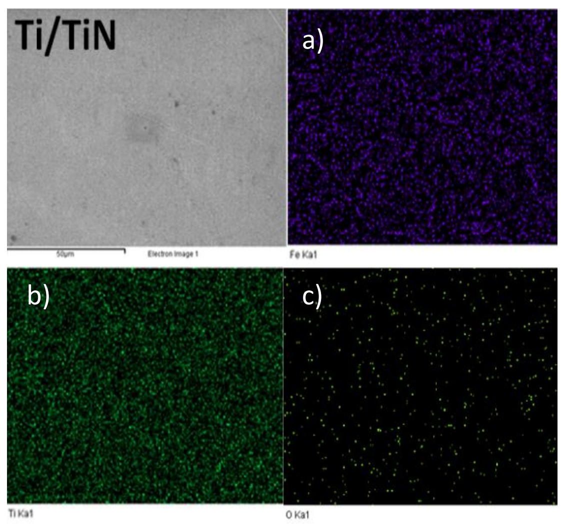

Figure 7 Analysis of elements of the 316L sample coated with Ti/Ti. a) iron, b) titanium, c) oxygen

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Conclusions

According to the results obtained during the synthesis and characterization of Ti/TiN thin films, obtained through the PVD process on a 316L steel, the tribological properties of the substrate were improved, this can correlate with an increase in the lifetime of mechanical elements. The Ti/TiN coating presented a lower coefficient of friction in wet conditions than uncoated substrate. The adhesion tests revealed a good adhesion to the substrate, no catastrophic detachment of the coating was observed when the indentation footprints were compared to the failure map of VDI 3198 standard.

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