1020 steel coated with Ti/TiN by Cathodic Arc and Ion Implantation

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Abstract. TiN coatings have been widely studied in order to improve mechanical properties of steels. In this work, thin Ti/TiN films were prepared by plasma based immersion ion implantation and deposition (PBII&D) with a cathodic arc on AISI 1020 steel substrates. Substrates were exposed to the discharge during 1 min in vacuum for the deposition of a Tiunderlayer with the aim of improving the adhesion to the substrate. Then, a TiN layer was deposited during 6 min in a nitrogen environment at a pressure of 3×10^{-4} mbar. Samples were obtained at room temperature and at 300 ºC, and with or without ion implantation in order to analyze differences between the effects of each treatment on the tribological properties. The mechanical and tribological properties of the films were characterized. The coatings deposited by PBII&D at 300 ºC presented the highest hardness and young modulus, the best wear resistance and corrosion performance.

1. Introduction

Cathodic arc coatings have been extensively studied and used due to their high deposition rates. In cathodic arc discharges a metallic plasma jet is ejected from the cathode surface and it is deposited onto a substrate. The substrate conditions used during the deposition process -temperature and bias respect to the plasma potential- influence on the structure, morphology and adhesion of the film [1,2].

Nanostructured Ti thin films have been obtained using cathodic arcs and also by combining plasma immersion ion implantation and deposition (PIII&D) [3, 4]. Polycrystalline titanium thin films have been widely employed as interlayer between the substrate and different coatings in order to improve adhesion strength, corrosion resistance and wear performance, as well as to promote the growth of crystalline phases of the coating.

Studies on the properties of TiN coatings obtained using PIII&D revealed significant improvement of tribological performance with the application of pulsed bias [5]. There are also studies on the tribological properties of TiN coatings grown with cathodic arc on stainless steel including Ti interlayers [6].

In this work, thin Ti/TiN films were prepared by PIII&D with a cathodic arc on AISI 1020 steel substrates. Coatings were obtained at different temperature and with or without ion implantation in order to analyze differences between the effects of each treatment on the tribological properties.

2. Experiment

The experiment was carried out in a d-c vacuum arc system, which is schematically shown in figure 1. The discharge was produced between a cylindrical Ti cathode (60 mm in diameter) and a grounded annular Cu anode (80 mm in diameter). Both electrodes were mounted on an insulating piece that set an electrode separation of about 15 mm. The cathode was surrounded by a floating shield. The discharge circuit consisted in a current supply (18 kW, 150 A) in parallel with a capacitor bank (165 mF) connected to the electrodes through a series inductor (2.8 mH) in order to provide arc stability. A high voltage generator applying pulses of 8 kV (30 μs, 200 Hz) was employed for ion implantation. The chamber was an electrically insulated stainless steel cylinder (25 cm long, 10 cm diameter). The base pressure of the system was lower than 10^{-5} mbar.
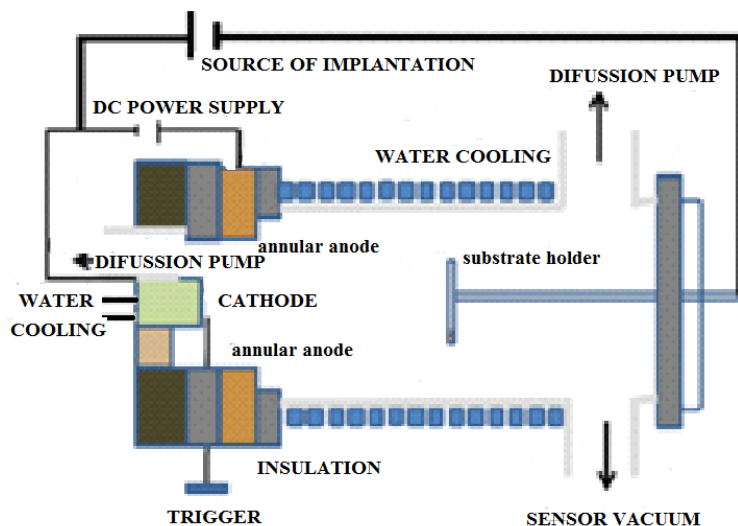

Figure 1. Schematic diagram of the d-c vacuum arc system.

The employed substrates were AISI 1020 steel sheets (22 mm long, 17 mm wide and 3 mm thick). They were placed at 20 cm from the cathode surface on a heater. The surfaces were previously cleaned with alcohol in an ultrasound bath for 30 min. Substrates were exposed to the discharge during 1 min in vacuum for the deposition of a Ti underlayer, and during 6 min in a nitrogen environment at a pressure of 3x10-4 mbar for TiN deposition. Samples were obtained at room temperature and at (300 ± 10) ºC, with and without implantation.

Film thickness was determined by using a Calotester, the coating was eroded with a steel ball (diameter 22.2 mm) employing standard metallurgical diamond lapping paste (1/4 μm). The tribological characterization was performed using a brand tribometer (CSEM Instruments SA) with an alumina ball at a speed of 5 cm/s with a load of 1 N for a distance of 400 m, in air at 18 ºC and 43% relative humidity. Nanohardness was measured with a nanoindenter Nanovea using the model of Oliver and Pharr and a maximum load of 5 mN. Corrosion rate measurements were performed by a potentiostat galvanostat PG-TEKCORR USB 4.1 with a brine solution (H_2O + NaCl 3.5%) and Ag/AgCl electrodes. The roughness was studied with a KLA Tencor ALPHA STEP 120 profilometer, using a diamond-tipped stylus, with a resolution of 10 Å.

3. Results and discussion

To identify samples prepared in each condition, coatings deposited at room temperature with cathodic arc are denoted as A, with cathodic arc and ion implantation as AI, and coatings obtained with arc and implantation at 300 ºC are named AIT.

Coatings obtained in different growth conditions were examined by a calotester and the thickness resulted in (740 ± 50) nm for all the samples. This value corresponds to the whole film (Ti + TiN), the thickness of each layer being not able to assess. Surface roughness results are shown in table 1. Roughness is very similar in all the films and is clearly lower than that of the substrate. Typical hardness measurements by the method of Oliver and Pharr are shown in figure 2. The figure shows

measurements at different places along an AI sample. It can be noticed an elastic/plastic deformation and that the signals are continuous, indicating the absence of film cracking during the indentation. Results obtained for hardness and elastic modulus from these measurements for different deposition conditions are listed in table 1.The substrate hardness measured with the Brinell hardness test was 130 HB (equal to 1,4 GPa) and the elastic modulus for the AISI 102 steel was ~ 200 GPa, both values were included in table 1 in order to compare them with the obtained results. It can be seen that the hardness and elastic modulus increased with the coatings, the obtained values being similar for different deposition methods. It is worth mentioning that the hardness measurement can be influenced by the hardness of the substrate, given that the depth is higher than 10% of the thickness.
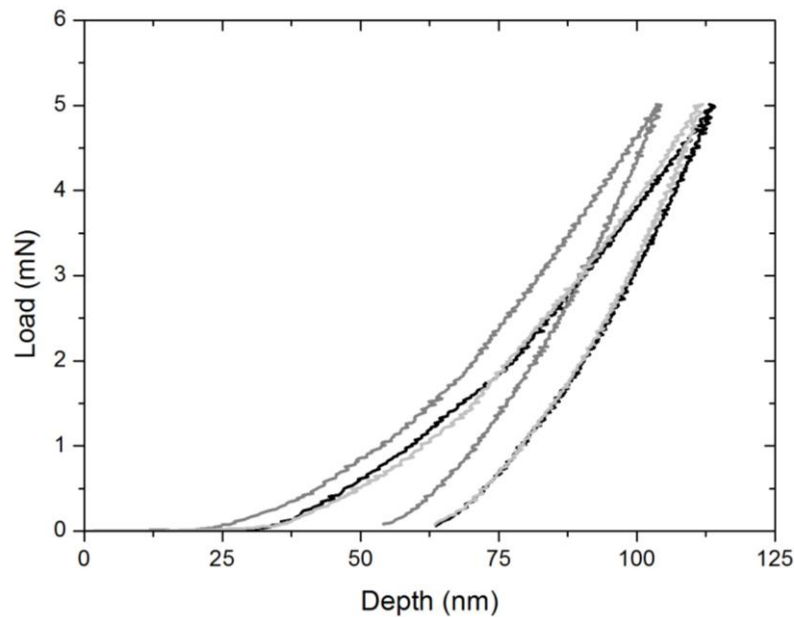

Figure 2. Hardness measurements at different places of an AI sample.

Table 1. Roughness, hardness, elastic modulus and wear rate for different growth conditions

Growth conditions	Substrate	A	AI	AIT
Roughness (± 2 nm)	624	189	194	202
Hardness (± 2 GPa)	1.4[a]	27	26	28
Elastic modulus (± 40 GPa)	200[a]	290	320	400
Wear rate (μm^3/Nm)	0.84	0.22	0.27	0.003

[a] Hardness and elastic modulus of the substrate are indicated as reference

Figure 3 shows wear measurements corresponding to an AIT sample. In the post wear scarring, no detachment or delamination can be observed. The wear rate obtained from the evaluation of the worn volume for different deposition conditions is presented in table 1. The wear rate decreases significantly, respect to the substrate, in samples A and AI, and even more for samples AIT.
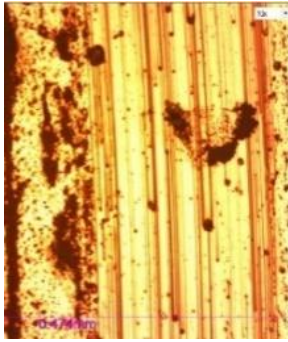
Figure 3. Wearscarring from an AIT coating.

Friction coefficient for Ti/TiN coating deposited in different conditions is plotted in figure 4. Signals corresponding to the coatings were registered up to 400 m distance, while that of the substrate was measured to 100 m. Samples prepared in the three conditions presented a similar behaviour, the friction coefficient being initially in the range 0.2 – 0.3 and finally reaching the substrate value, around 0.8, indicating an abrasive process. It can be seen that the transition from the initial to the final value for the friction coefficient takes places at longer distance in the cases of the samples AIT and A.
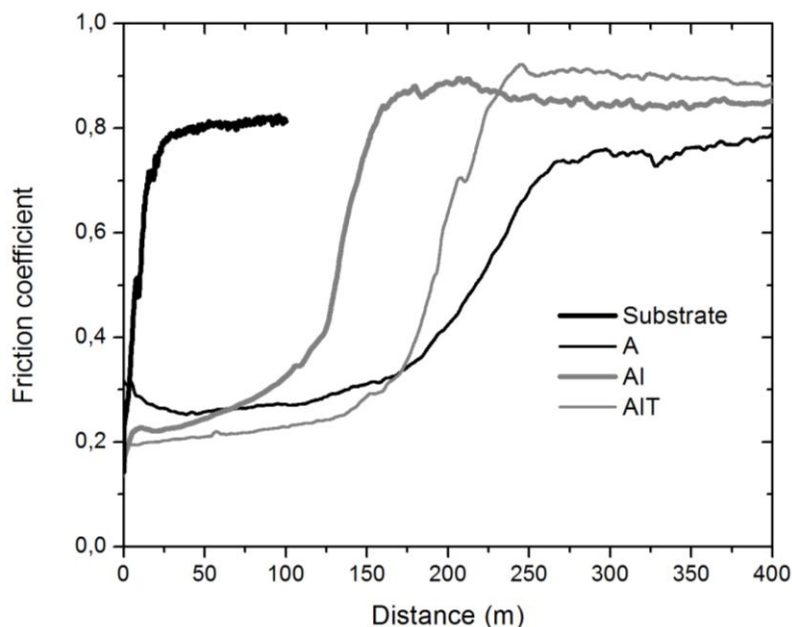

Figure 4. Friction coefficient for Ti/TiN samples deposited in different conditions.

Figure 5 shows the corrosion potential as a function of the current density for samples deposited in different conditions. It can be clearly noticed that the sample prepared with arc and ion implantation at 300 ºC (AIT) exhibited the lowest corrosion potential, while samples grown in other conditions presented similar values to that of the substrate. Results obtained from corrosion measurements are summarized in table 2. The AI sample presented similar corrosion behaviour to that of the substrate, regarding all the parameters. On the other hand, the AIT coating showed the lowest corrosion velocity, and then the best corrosion resistance performance.
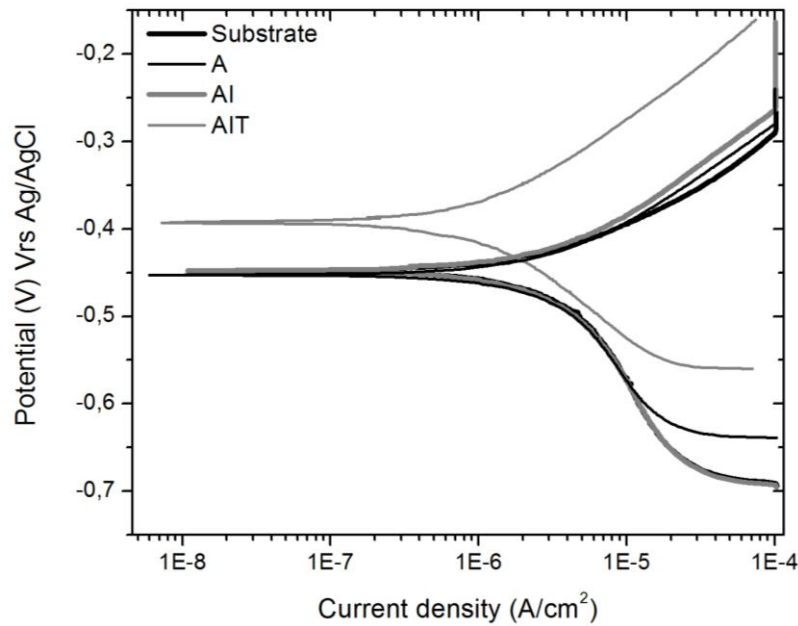
Figure 5. Corrosion potential as a function of the current density for Ti/TiN samples deposited in different conditions.

Table 2. Corrosion parameters obtained from Ti/TiN samples deposited in different conditions.

Growth conditions	Corrosion velocity (mm/y)	Current density (nA/cm^2)	Corrosion potential (mV)
Substrate	164	320	-448
A	128	249	-453
AI	163	318	-448
AIT	119	231	-393

4. Summary

Ti/TiN coatings on stainless steel substrates were obtained by cathodic arc deposition and ion implantation. The obtained hardness and friction coefficient was similar for all the samples. Surface hardness of the coated samples was one order of magnitude greater than that of the substrate. Despite the final friction coefficients were similar for the different coating conditions, for A and AIT samples the transition from the initial low value to the final value for the friction coefficient takes places at longer distance.

The best corrosion and wear properties were obtained for AIT samples. The corrosion velocity for AIT samples was ~ 30% lower than that of the substrate, while the reduction in the corrosion velocity for A samples was ~ 20%. The wear rate in all deposition conditions was significantly reduced respect to the substrate, the value corresponding to AIT samples being two orders of magnitude lower than those of the other conditions.

Therefore, deposition by cathodic arc combined with ion implantation and heating the substrate led to the best overall performance and is promising for industrial applications.

5. Acknowledgments

6. References

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