Optimization of a PIII&D System Using a Cathodic Arc with Titanium

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Abstract. A plasma immersion ion implantation and deposition (PIII&D) system was recently built at INFIP. A dc cathodic arc with a Ti cathode of 5 cm in diameter and an annular anode of 8 cm in diameter was employed as the plasma source. The substrate chamber was electrically insulated and connected with the main discharge chamber through a straight magnetic duct. The discharge current was run at 100 A. The substrate was biased with a pulsed generator (30 kV, 30 A, 0.05 - 3 kHz) based on a pulse transformer controlled by IGBT switches. In this work the optimization of the process as function of the pulse parameters is presented. The characteristics of Ti coatings on steel substrates obtained varying the pulse amplitude from 2 to 12 kV and the pulse frequency from 200 Hz to 400 Hz were analyzed and compared with films grown without biasing the substrate. The thickness was determined weighting the samples before and after the treatment. The morphology was observed with an atomic force microscope. The film structure was studied by x-ray diffraction.

1. Introduction

Plasma immersion ion implantation combined with deposition (PIII&D) using cathodic arcs as plasma sources is a technique that has demonstrated to be useful for the fabrication of nanostructure coatings. Applying PIII&D, the substrate is immersed into the plasma plume produced with a cathodic arc and is biased with high-voltage pulses. When the high negative potential (typically many kilovolts) is on, a plasma sheath forms around the workpiece and ions are accelerated towards the surface and implanted. While the bias is off ions condense on the surface resulting in the deposition of a coating or a film. The use of high voltage pulses to bias the substrate allows to obtain denser films and to relax internal stress improving the adhesion, tribological and mechanical properties [1].

In this work the optimization of the process as function of the pulse parameters is presented. The characteristics of Ti coatings on steel substrates obtained varying the pulse amplitude from 2 to 12 kV and the pulse frequency from 200 Hz to 400 Hz were analyzed and compared with films grown without biasing the substrate.

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2. Experimental setup

A plasma immersion ion implantation and deposition (PIII&D) system was recently built at INFIP, which is shown schematically in figure 1. A dc cathodic arc with a Ti cathode of 5 cm in diameter and an annular copper anode of 8 cm in diameter was employed as the plasma source. A tungsten striker was brought into contact with the cathode surface and later removed to trigger the discharge which was run continuously at 100 A. 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. Both cathode and anode were refrigerated during the arc discharge by a water cooling system by means of a centrifuge pump with a nominal discharge of 50 l/min. The substrate chamber was electrically insulated and connected with the main discharge chamber through a straight duct. One vacuum system, composed of mechanical and diffusion pumps, pumps separately the plasma generation and deposition chambers to a base pressure of less than 10-4mbar. The pressure was measured by means of an Active Pirani Gauge (Edwards APG-L-NW16).

The substrate was biased with a pulsed generator (30 kV, 30 A, 0.05 - 3 kHz) based on a pulse transformer controlled by IGBT switches, shown in figure 2. The pulse amplitude varied from 2 to 15 kV and the frequency from 100 to 300 Hz. A high voltage probe (Tektronic P6015A, 1000X, 3.0 pF, 100 MΩ) was used to measure the pulse parameters previous to and during the arc discharge.
Most of the samples were made of AISI 316 steel and placed 25 cm and 40 cm from the cathode. The exposure time to the deposition process was 4 minutes. Some films were grown on silicon boron doped substrates with one side polished, in order to use a smooth surface for morphology characterization of the film. The samples were weighted prior to and after the coating. The crystalline structure of the films was identified by X-ray diffraction (XRD) using a Philips PW 3710 diffractometer with a CuKα source. It was operated with glancing angle geometry by using a Philips thin film attachment, with an angle of incidence of 1 deg. The film morphology was studied by tapping mode atomic force microscopy (AFM) with a Nanoscope III Digital-VEECO.

3. Results

The deposition rate \( r \) is defined as the thickness of the coating over the time of exposure to the arc discharge. The thickness was determined from the deposited mass on the substrate taking the density value as the Ti bulk density (4.507 g/cm³). The deposition rate studied as a function of the pulse amplitude, varying the distance between the cathode and the substrate, is shown in figure 3. Changing the axial position from 25 to 40 cm \( r \) diminished to a 40 %, leading to a loss of accuracy in its determination for the samples placed further from the cathode. On the other hand, the deposition rate did not show a significant dependence on the pulse amplitude and presented a tendency to increase with the frequency of the pulse. This fact suggests that the contribution of sputtering to the deposition rate is not significant for the employed voltages and the increasing with the frequency could be attributed to an improvement on the ion sticking coefficient when the high voltage pulse is on.

![Figure 3. Deposition rate as a function of the pulse amplitude for different distances between the substrate and the cathode.](image)

The dependence of the rate deposition with the pulse frequency was also investigated. The substrates were placed 25 cm from the cathode with pulse amplitude of 8 kV. The deposition rate versus the frequency of the pulse is shown in figure 4. The deposition rate presented a slight increase with the frequency of the pulse.
Figure 4. Deposition rate as a function of the frequency of the pulse for a pulse amplitude of 8 kV at a distance of 25 cm between the substrate and the cathode.

AFM images of films grown on silicon substrate are presented in Figure 5. The morphology of films obtained by the PIII&D technique (figure 5 a)) was compared with the surface of titanium coatings deposited without implantation (figure 5 b)). The surfaces of the Ti coatings with and without implantation did not present significant differences; both films were very compact and presented a columnar growth. The surface profiles of the films obtained from AFM images are shown in figure 6. From these profiles, the column size was found in the range 20 – 40 nm; the roughness was estimated in 0.23 nm.

XRD registers obtained from films deposited at 25 cm from the cathode with a pulse frequency of 200 Hz and with different voltages are shown in figure 7. The main peaks of Ti were observed in all films, but in difractograms corresponding to PIII&D a broadening of Ti peaks was also observed. The changes in the difractograms can be attributed to the Ti implantation, this process could introduce titanium in the crystal lattice modifying the interplanar distances in the film.
Figure 6. Surface profiles obtained from AFM images of films: a) deposited by PIII&D and b) deposited without implantation.

Figure 7. Diffractograms of Ti coating without implantation and Ti coating by PIII&D technique for different pulse amplitudes.

4. Conclusions
The deposition rate with implantation did not present significant differences from the deposition rate without implantation, then sputtering was not affecting the treatment. The increase of the deposition rate with the frequency suggested an improvement on the ion sticking coefficient with the pulsed bias for implantation. The PIIID technique did not introduce significant changes in the morphology of the films, obtaining very dense structures, with a columnar growth and a low roughness. However, PIII&D affected the film structure introducing changes in the interplanar distances of the crystal lattice.

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References
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