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Magnetic-field enhanced plasma immersion ion implantation and deposition (PIII&D) of diamond-like carbon films inside tubes

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ABSTRACT

The present work deals with Diamond-like Carbon (DLC) films deposition inside metallic tubes using magnetic field generated in a PIII&D system. Firstly, the features of plasma discharges with the magnetic field application were studied using different feeding gases as nitrogen, methane and acetylene. The experimental results demonstrate that a stable hollow cathode discharge can be established inside the tubes as the magnetic field is applied. The discharge breakdown is strongly affected by the presence of the magnetic field during the treatment. Secondly, PIII&D experiments regarding DLC films deposition inside the inner surface of the tubes are also described and those ones enhanced by the applied magnetic field are emphasized. The sample tubes used in both experimental stages are of austenitic stainless steel with 150 mm in length and different diameters: 110 mm, 40 mm and 20 mm. For the case of DLC deposition, polished steel samples were fixed in the bottom of the inner tube wall for subsequent analysis of the coatings. The as-coated DLC samples surface were analyzed by Raman Spectroscopy, X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM), optical profilometry and also pin-on-disk tests. The properties of the obtained DLC films are correlated to the magnetic field used during the PIII&D process. In this work, the optimal range of magnetic field intensity is shown for depositing DLC films with acceptable adhesion strength on the inner surface of steel tubes.

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1. Introduction

Diamond-like carbon (DLC) films are interesting multifunctional coatings for deposition on metallic surfaces which are applied in several modern technologies. Amongst other outstanding properties of DLC films, their remarkable resistance against corrosion and wear, high hardness, low roughness, and also very low friction coefficient make them good candidates to be used in many fluid transport applications, acting as abrasion-resistant coatings in the inner surface of tubes [1,2]. In some special applications, high quality technologies are needed to modify the inner surface of metallic tubes and pipes, the inside of cavities or holes and cylindrical components. In the case of petroleum and gas industries, for example, large conductive pipes have to withstand the corrosive wear due to the deposition of salt scales on the internal metallic surface. Besides, the installation environment can also cause an aggressive wear of its external surfaces [3–5]. Moreover, in the other limit of tube size, very small bore tubes like that ones used in propulsion and thermal control systems of satellites have to resist corrosion caused by the fuels and the cooling fluids, respectively [6,7]. In all cases cited above, highly durable surfaces are demanded and thus, a long lifetime of those components would be expected as a result of the use of protective coatings.

In this context, publications related to DLC deposition inside tubes have increased recently, despite the limitations that are found in reproducing some film-substrate pairs with properties that match their real application conditions. This task is even more difficult when using plasma treatments, especially those ones combined with plasma based ion implantation [8–12]. The extensive scientific literature concerning inner-tube coatings is generally aimed to overcome the problem of film inhomogeneity and their deposition inside slender and millimetric tubes. Recently, DLC films have been deposited using a condition of low gas pressure with high voltage pulse or at high gas pressure with relatively low bias applied to the metallic tube. Indeed, very specific discharge conditions are required to run PIII&D in cylindrical substrates: at higher working pressures the mean free path lowers and, in such a way, enables an efficient deposition; in addition, high pressure operation limits the applicable maximum high voltage pulse due to the occurrence of arcs. On the other hand, for low gas pressure, the discharge

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condition is more controllable and higher voltage pulses can be applied to the substrate tube [5,10,11].

As well known, for the improvement of plasma processing technologies, studies to increase the plasma confinement and to obtain high density plasmas are of great interest. For this reason, using stable and high density magnetically confined plasmas, can contribute to enhance film deposition inside tubular substrates. Other prospects arising from the use of such high density plasmas are the application of batch process to simultaneously treat many substrates, and the modification of long pipes like those used in the industry [5,12].

In the last decade, special efforts have been made to solve the practical problems related to the modification of inner surfaces of tubes. In previous papers, treatments with *ExB* fields have been used in PIII experiments to implant nitrogen inside stainless steel tubes [13]. There, an intense background gas ionization was provided by the trapped electrons, which drifted in the crossed field lines with velocity given by $v_{ExB} = ExB/B^2$. The resultant increase in plasma density by such a scheme, has also contributed to enhance the deposition of carbon ions onto flat steel substrates [14]. But, under similar configuration, the contribution of the magnetic field to carbon deposition inside tubes has not been yet investigated.

In this work, such particular method which combines PIII&D of DLC with magnetic field confinement of the plasma is described. By using this combination, the improvement of the plasma confinement inside hollow steel cylindrical substrates is shown. Therefore, firstly, this work is aimed at the evaluation of discharge breakdown in nitrogen, methane and acetylene gases influenced by crossed electric and magnetic fields. Secondly, some typical characteristics of the as-coated DLC films obtained inside the steel tubes in such configuration will be shown.

2. Experimental details

The PIII&D processing employed here was carried out using an experimental system from the Associated Laboratory of Plasma at INPE. It consists of a cylindrical stainless steel vessel of 20 l (260 mm diameter and 380 mm length) equipped with a double set of magnetic coils. Its detailed description is found in some recent published paper of our research group [15].

The study performed herein is concerned with a specific procedure for DLC deposition using crossed electric and magnetic fields. The attained results are compared for each group of substrate tubes, and the improved properties in each analyzed case are pointed out.

Three experimental cases were carried out in which a large, middle and small cylindrical sample tubes (AISI SS304) were used. They were classified according to their outer diameter: group I (110 mm), group II (40 mm) and group III (20 mm). The length and wall thickness of the substrate tubes were fixed at 150 mm and 2 mm, respectively. SS304 alloy was chosen as substrate because of its wide range of applications in industry, low cost, availability and well-known properties. The inner surface finishing of tubes was kept as-received from machining, exhibiting an arithmetic average roughness of around 344 nm. These sample tubes were ultrasonically degreased in a detergent solution and after that in isopropyl alcohol for 15 min.

At the first place, the discharges in plasmas were evaluated for each group by adjusting the high voltage (H.V.) pulse, the gas working pressure, and the applied magnetic field. In the presently discussed cases, the plasma ignition was achieved by only applying H.V. pulses to the tube. The applied voltage pulse and obtained current were provided by a RUP4 power supply and were recorded by a digital oscilloscope Tektronix model TDS360. Pictures of the plasma from inside the tube were taken through the glass window and compared for different conditions.

By using the arrangement of coils of our system, the maximum magnetic field intensity was 110 G in the center of the chamber. The magnetic field produced inside the vacuum chamber was mapped in the axial direction using a Gaussimeter Walker model MG3D. The probe position corresponded to the center of the chamber where the magnetic trap has its minimum value. Within the possible operational range, the magnetic field intensity was classified as low (0 G–40 G), moderate (41 G–70 G) and high (71 G–110 G). The negative high voltage pulses were varied between 3.5 and 7.5 kV, with 20 μs width and repetition rate of 500 Hz, and the working pressure was varied from 2 Pa to 5 Pa in this experimental stage. The magnetic field intensity and the supplied gas pressure were the only parameters changed.

In the second place, the deposition experiments were run by using the H.V. pulse and the magnetic field strength as the settled processing parameters. In the third group (the smallest diameter tube), the film had to be deposited using a working pressure about ten times higher than in the other groups to reach appropriate discharge conditions. These parameters used for DLC deposition are shown in detail in the Section 3.2. In this second step, some polished SS304 samples (15 mm diameter and 2 mm thickness) were placed on the bottom of the inner wall of the tubes, at the center part of the axial direction, for subsequent analysis of the DLC coating. As we know, DLC films keep the same topography of the substrate. So, the substrates were polished to minimize their interference when the properties of the deposited films were analyzed. They were polished to a mirror-like finish with diamond paste of 3 μm grain size, and presented arithmetic average roughness (Ra) of around 16 nm. Then they were cleaned in ultrasound bath with isopropyl alcohol for 15 min and dried in a nitrogen atmosphere. In the third group, small strips of silicon samples (4 mm × 15 mm) were used because of the limited space for the insertion of steel samples inside the tube.

Chemical structures of the films deposited on samples placed inside the tubes were measured by a Raman Spectrometer Horiba Scientific (LabRam HR Evolution model). The measurements were made in air at room temperature using Ar^+ laser ($\lambda = 514.5$ nm) and Labspec6 software. Their Raman signature was composed of two broad bands, namely D and G peaks, centered around 1360 cm^{-1} and 1580 cm^{-1}, respectively, in the visible excitation range [16]. The spectra were deconvoluted in two Gaussian functions by using the Fityk 0.9.7 and Origin softwares.

The XPS chemical analysis of the DLC coated surfaces were performed on a Kratos Axis UltraDLD Electron Spectrometer, using X-Ray Monochromator Al kα (1486.6 eV) radiation. The experimental resolution of the binding energy was less than 0.5 eV. The samples were excited with monochromatic X-rays with the X-ray source operated at 20 kV acceleration voltages and with an emission current of 10 mA. XPS spectra were collected in ultra-high vacuum at 1.7×10^{-8} Pa, in the sample analysis chamber. Survey-scan spectra were obtained with 160 eV band-pass energy, while the C 1s, N 1s, O 1s and Si 2p regions in high-resolution core level spectra were taken in the constant analyzer energy mode with band-pass energy of 40 eV and energy step of 0.1 eV. Binding energies were corrected for possible charging effects by referencing to the adventitious C 1s line set at 284.8 eV using the origin software.

Friction coefficients were measured by means of a CSM-Instrument pin-on-disk tribometer, under 5 N load using an alumina ball (1800 HV) of 3 mm diameter, linear speed of 5.0 cm/s, 5.0 Hz of acquisition rate, 5000 revolutions, humidity of 67%, and 1.0 mm of wear track radius. DLC thickness was measured by a Wyko NT1100 optical profiler at the film-substrate transition area. For the third group, the obtained thickness of DLC could be estimated from SEM cross-section images of cleaved Si samples. The worn tracks on DLC film surfaces were observed by SEM and optical profilometer. In the case of an uncoated steel sample, it was not possible to see the track using the optical profilometer (maximum scan length of 301 μm).

3. Results and discussion

3.1. Evaluation of the discharge breakdown

The characteristics of the discharge breakdown were observed for each experimental group after applying the magnetic field. The selected

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