XPS INVESTIGATION OF THE CHEMICAL PROPERTIES OF SS 304 TREATED BY NITROGEN PIII INSIDE A CONDUCTIVE TUBE

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1. Introduction

Plasma immersion ion implantation (PIII) is a widely used technique for the surface modification of materials, looking mainly for improvements of mechanical, electric or magnetic properties of complex-shaped three-dimensional objects [1,2]. It has been extensively used to improve wear resistance and hardness of stainless steel (SS). However, the success of the treatment is closely related with the implantation of a high dose of ions and/or the achievement of a moderate temperature of the substrate during the process. Besides the plasma parameters, the chemical composition of the substrate strongly influences the characteristics of the treatment and modification of the treated surface. X-ray photoelectron spectroscopy (XPS) has been much used for the investigation of the chemical changes occurring on the near-surface region. It reveals which chemical elements are present at the surface and it informs us about the chemical bound nature which exists between these elements. An appropriate data processing leads also to the specimen elemental composition. In addition, XPS spectra reflect atomic scale chemical interactions, i.e. the bonds between neighboring atoms. In order to determine the effect of implantation inside the tube in presence of a magnetic field, stainless steel (SS) samples were implanted with nitrogen ions with and without the presence of auxiliary electrode (AE). Samples treated under these two conditions demonstrated a remarkable difference in terms of chemical properties.

2. Experimental

In this experiment, tubes with 110 mm diameter and 150 mm length were placed in a large chamber of 600 liters in order to carry out PIII treatments. Polished SS 304 samples with 0.5 mm thickness and 0.75 mm radius were placed inside tube and treated during 60 min to analyze the effects of PIII in E x B fields on the tube inner wall. Two arrangements were used in presence of magnetic field: (1) tube without AE and (2) tube with a grounded AE. The nitrogen PIII treatment conditions were kept constant in voltage of 6 kV, 500 Hz frequency, 20 µs pulse length, current density during implantation of 7.3 mA/cm^2. In this system, the breakdown of the discharge is facilitated by presence of the AE and the magnetic field at gas pressure above $2x10^{-2}$ mbar [3]. To monitor the temperature of the tube an infrared thermometer Mikron model M90 was used with nominal range between 250 and 2000 ^{o}C. After treatment, surface chemical characterization of the samples was carried out by X-Ray Photoelectron Spectroscopy by a Kratos Axis Ultra^{DLD} Electron Spectrometer, using X-Ray Monochromator Al kα radiation. The experimental resolution of the binding energy was less than 0.5 eV. Survey-scan spectra were made at a pass energy of 160 eV, while for C 1s, N 1s, O 1s, Fe 2p and Cr 2p individual high-resolution core level spectra were taken in the constant analyzer energy mode with a 40 eV pass energy and a 0.1 eV energy step. After background subtraction, peak deconvolution was performed using the CasaXPS software. The chemical state of SS 304 was tested by examining the Fe 2p and Cr 2p signal. The nitrogen implanted on the steel surface was monitored by means of the characteristic C 1s, O1s and N 1s peaks.

3. Results and Discussions

The effects of nitrogen incorporation after plasma treatment on the chemical properties of SS 304 were studied. The XPS results revealed the presence of nitrogen adsorbed on SS 304 and the well known phases such as Fe_xN and CrN. The Fe 2p spectrum showed a band at about 710.5 eV, corresponding to iron-oxide, evidencing the existence of a thin oxide layer on the sample. Higher intensity peaks of expanded austenite were detected by XPS for samples treated in discharges with the presence of the AE. This is a consequence of higher concentration of nitrogen implanted into SS 304, confirming the efficiency of the PIII process inside the tube when using the auxiliary electrode.

4. References

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