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Plasma immersion ion implantation (and deposition) inside metallic tubes of different dimensions and configurations

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ABSTRACT

There is a strong need for developing methods to coat or implant ions inside metallic tubes for many practical contemporary applications, both for industry and science. Therefore, stainless steel tubes with practical diameters of 4, 11 and 16 cm, but short lengths of 20 cm, were internally treated by nitrogen plasma immersion ion implantation (PIII). Different configurations as tube with lid in one of the ends or both sides open were tested for better PIII performance, in the case of smallest diameter tube. Among these PIII tests in tubes, using the 4 cm diameter one with a lid, it was possible to achieve tube temperatures of more than 700 °C in 15 min and maintain it during the whole treatment time (typically 2 h). Samples made of different materials were placed at the interior of the tube, as the monitors for posterior analysis, and the tube was solely pulsed by high voltage pulser producing high voltage glow discharge and hollow cathode discharge both driven by a moderate power source. In this experiment, samples of SS 304, pure Ti, Ti6Al4V and Si were used for the tests of the above methods. Results on the analysis of the surface of these nitrogen PIII treated materials, as well as on their processing methods, are presented and discussed in the paper.

1. Introduction

Recent interests of the plasma immersion ion implantation (PIII) and Deposition (PIII&D) community comprise ion implantation of nitrogen or other species and deposition of Diamond Like Carbon (DLC) and other films, inside metallic tubes [1–4]. These are difficult tasks for beam ion implantation user but relatively simple to be performed with plasma immersion technique. Possible applications of the PIII and PIII&D in metallic tubes include: petroleum extraction tubes deposited with DLC (many kilometers long), and already being tested in offshore, mainly in pre-salt basins [4]; nitrogen implantation in tubes for iron ore transport in long ore ducts (more than 500 km, not yet treated) [5]; recovery of old organ pipes and their reinforcement [6]; small to medium size rocket propulsion nozzles implanted with nitrogen with high temperature PIII [7]; internal protection of liquids transporting pipes in fusion and fission devices [8]; and so on.

Deposition of DLC in large and long tubes has already been demonstrated by Wei and coworkers [4], while in small bore tubes of diameter with millimeter sizes, DLC deposition was successfully obtained by Baba and coworkers [3]. More recently, 3–4 cm diameter metal tubes were coated with DLC and further improvements in that layer have been attained by additional ion implantation [9]. Nitrogen implantations in many practical size metal tubes are required for the above mentioned applications. It is also a step in the deposition of DLC film inside tubes [10] to avoid its delamination.

Therefore, stainless steel tubes with practical sizes with outer diameters of 4, 11 and 16 cm, and short length of 20 cm and thickness of 2 mm, were internally treated by nitrogen PIII. Scaling to longer tubes will be attempted in the future. Different configurations as tube with lid in one of the ends or set-up with both sides open (all of them in laying down position) were also tested for better PIII performance.

Samples made of different materials were placed at the interior of the tube, as the monitors for posterior analysis, and the tube was solely pulsed by high voltage pulser producing high voltage glow discharge and hollow cathode discharge, both driven by a moderate power source.

Both, plasma or gas nitriding, are possible in SS304 workpieces in industrial scale nowadays. Tubes of different sizes made of that material are also coated internally by DLC using CVD or PVD

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plasmas [11]. However, treatments of such tubes by nitrogen implantation in an industrial scale are less common.

In the case of Ti or Ti alloys, ion nitriding at temperatures of up to 900 °C has been successfully attained in flat pieces by glow discharge nitriding [12]. Such high temperatures are necessary for strong diffusion of nitrogen inside the matrix of Ti or Ti alloys. However, in concave geometries, nitriding of such materials continues to be a challenge.

Successful nitrogen incorporations inside SS304 tubes of 11 cm∅ have already been demonstrated in the previous papers [13,14]. In those cases, convenient tube temperatures of about 350–400 °C were obtained and also in the SS304 samples placed at the interior wall of the tube used as monitors, during the nitrogen PIII treatment. The procedure to use monitor samples is taken because curved surfaces (from the tubes) are impossible to be used for many standard analysis purposes. Such a range in temperature allowed a strong diffusion of nitrogen into the SS304 lattice of both, samples and tube interior, during the PIII process, which resulted in quite thick nitrogen rich layers on them (3–5 μm). In those experiments, the emphasis were on the treatment of parts of the workpieces attached to the tube, as well as, on the studies of different positioning and configurations of the tube, mostly in the standing up position [13–15].

In the present experiment, we were additionally able to test the PIII process in a larger tube of 16 cm∅ and in a smaller tube of 4 cm∅. The larger diameter tube would be adequate as a fixture for larger components than ones treated previously in 11 cm∅ tubes, or in larger number of components as a batch processing method [13]. Using the tubes with 4 cm diameter was expected to lead to PIII with higher treatment temperatures, if similar power used for larger tubes was applied to it, which was confirmed by the results shown in this paper. By reaching more than 700 °C in such tubes, it is now possible to treat samples and components (including tubes) made of Ti or Ti alloys by nitrogen PIII. Results on the analysis of the treated samples surfaces in such dimensions and configurations are discussed here.

2. Experimental methods and set-ups

The PIII system that was used in this experiment is similar to the one used in reference [13]. Its general operation steps are described there but in the present case, the major difference to the previous PIII set-ups was that all the SS304 tubes were connected directly to the high voltage pulser output as before but used in the laying down position only. That allowed to comparing directly cases with tubes with a lid and without. No additional plasma source was used. The hollow cathode and the high voltage glow discharges inside the tubes were driven solely by the high voltage pulser. The metallic tubes were insulated from ground by a set of dielectric insulators (corrugated alumina tubes, then a glass

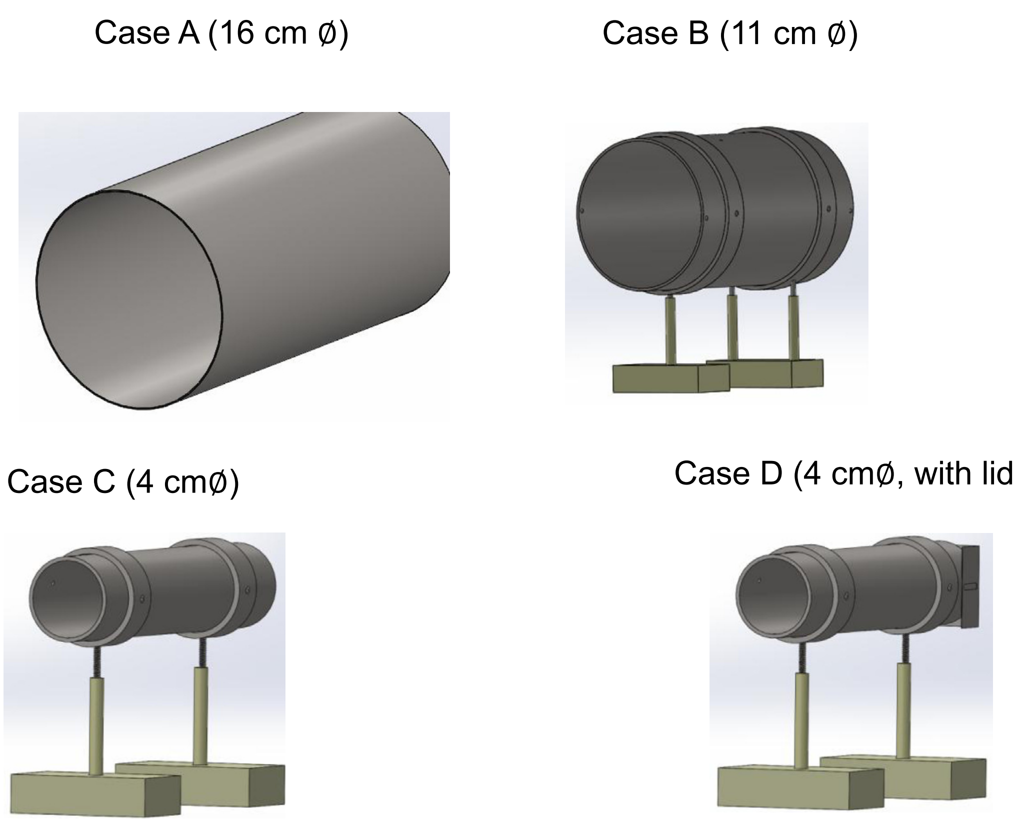

Fig. 1. Used Me-tube lying down configurations in this experiment.

Table 1
Optimized operation conditions of nitrogen PIII tests and treatments for different size tubes and configurations.

Tube configurations	Tube diameter (cm)	Pressure of N_2 (mbar)	Pulse length (μs)	Frequency (kHz)/primary current (A)	Peak pulse voltage (kV)	Peak pulse current (A)	Treatment time (min)	Final temperature
Case A	16	2.7×10^{-2}	30	1/100	3.5	5	15	<250 °C
Case B	11	4.6×10^{-2}	30	1/100	2	4	120	390 °C
Case C	4	4.7×10^{-2}	30	1/100	6	3	120	580 °C
Case D (w/lid)	4	1.5×10^{-2}	30	3/100	4	2.0	120	715 °C

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