Available online at www.sciencedirect.com
ScienceDirect
Surface & Coatings Technology 201 (2007) 7292–7302
SURFACE & COATINGS TECHNOLOGY
www.elsevier.com/locate/surfcoat

Titanium alloy surface treatment using an atmospheric plasma jet in nitrogen pulsed discharge conditions

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Received 20 February 2006; accepted in revised form 25 January 2007
Available online 3 February 2007

Abstract

This experimental work deals with the surface treatment of TiA6V4 titanium alloy using an atmospheric pressure plasma jet generated by a DBD discharge in nitrogen flow.

Firstly, the atmospheric pressure DBD reactor is described and the energy injected into the gap under chopped sinusoidal HV excitation is defined. Then, the influence of several experimental parameters on surface treatment is studied using several macroscopic characterization methods such as wettability with water drops and the ink test method. Results of XPS analysis on the treated samples are finally presented, offering insight into the microscopic modifications inflicted on the metallic surface by the afterglow treatment.

Experimental results presented in this work show that these particular conditions of HV excitation seem to be interesting from an application point of view. Based on mainly macroscopic surface diagnostics, it is shown that the electrical energy injected into the DBD reactor, as well as the characteristics (duty cycle) of the HV chopping signal influence the treatment results.

Keywords: Pulsed plasma; DBD; Atmospheric pressure; Nitrogen; Titanium alloy; Photoelectron spectroscopy

1. Introduction

Industrial applications involving surface treatment are numerous, extending over a large variety of materials like polymers, wood, ceramics, and metals [1–10]. Traditional methods for surface treatment are mostly chemical: materials are immersed in chemical baths which often exhibit hazardous and pollutant by-products. In particular the TiA6V4 titanium alloy, widely used in the aeronautical industry, often undergoes specific surface treatments for improving its adhesion and painting properties. For this alloy, typical treatment methods are mechanical and/or chemical or even electrochemical. To this day, the method the more frequently used is chromic anodic oxidation [11].

Several years now, the cold plasma technology has emerged as an alternative solution to surface treatment, having the advantage of being environmentally friendly. Low pressure plasmas meet the typical industrial demand for homogeneous surface treatment. However, the installation/maintenance cost of vacuum systems as well as the limits that these systems impose on the dimensions of the treated objects is a major drawback of this technology, especially in the case of low cost materials. On the other hand, atmospheric pressure plasmas tend to exhibit a filamentary character which limits the treatment's homogeneity. In the high pressure case as well, the treated objects' size is limited by the inter-electrode gap dimensions.

In this work an atmospheric pressure DBD (Dielectric Barrier Discharge) system, conceived for the treatment of materials in spatial afterglow conditions, is applied on TiA6V4

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0257-8972/$ - see front matter
doi:10.1016/j.surfcoat.2007.01.045

samples. Since the treatment takes place outside the reactor (spatial afterglow), there are practically no limitations in the dimensions nor the geometry of the treated objects. Furthermore, the condition of homogeneity is met by controlling the gas flow and is thus independent of the discharge's spatial homogeneity. When working under high gas flow conditions, the reactive species like metastables with long lifetimes, created during the discharge, can be transported outside the reactor and towards the material's surface. In the literature, the term "plasma jet" is now currently used to define these new plasma sources offering the possibility to surmount the technological barrier of the vacuum [12–15]. It must be understood that the "plasma jet" implies treatment in spatial afterglow conditions where the transported species are mainly neutrals since charged particles rapidly recombine outside the plasma source [16].

In general, surface treatment when applied to metallic alloys can be thought to consist in affecting three distinct stages of physico-chemical modifications. The first stage is surface *cleaning*: the removal from the surface of oils and other organic lubricants used during the samples' manufacturing and cutting process. Secondly, the superficial native oxidation layer has to be removed by *etching*. Finally, surface *chemical conversion* could be sought, giving the final state of the surface. The aim of this work is to investigate, depending on experimental conditions, the efficiency of the "plasma jet" on achieving these distinct but closely related and overlapping stages of treatment. According to the initial nature of the metal (physical and chemical states) and the experimental parameters of the "plasma jet", the aforementioned treatment phases (cleaning, etching and chemical conversion) could be necessary for industrial applications like painting and adhesion.

In the following part experimental details are given for the atmospheric pressure DBD reactor and the titanium alloy used. In the third part experimental results obtained on the titanium alloy surface treatment using different macroscopic and microscopic (XPS) analyses are presented. A brief discussion on surface treatment results and on the possible active species present under the plasma jet conditions here investigated is the object of Section 4. Conclusions are given in the Section 5 final section of this work.

2. Experimental details

2.1. Experimental device

The experimental device used in this study is shown in Fig. 1 and has been described in detail in previous works [17,18]. It is composed of three components summarized below: the electrical circuit, the flow system and the treatment vessel.

The power generator provided by AcXys Technologies electrically supplies the DBD reactor via coaxial HV cables. The DBD industrial reactor (S60 of AcXys Technologies, Grenoble, France) is in a coaxial cylindrical electrode configuration. The HV signal is applied to the inner electrode which is covered by a dielectric while the outer electrode is grounded. A 1/1000 voltage probe (Tektronix P6015A,

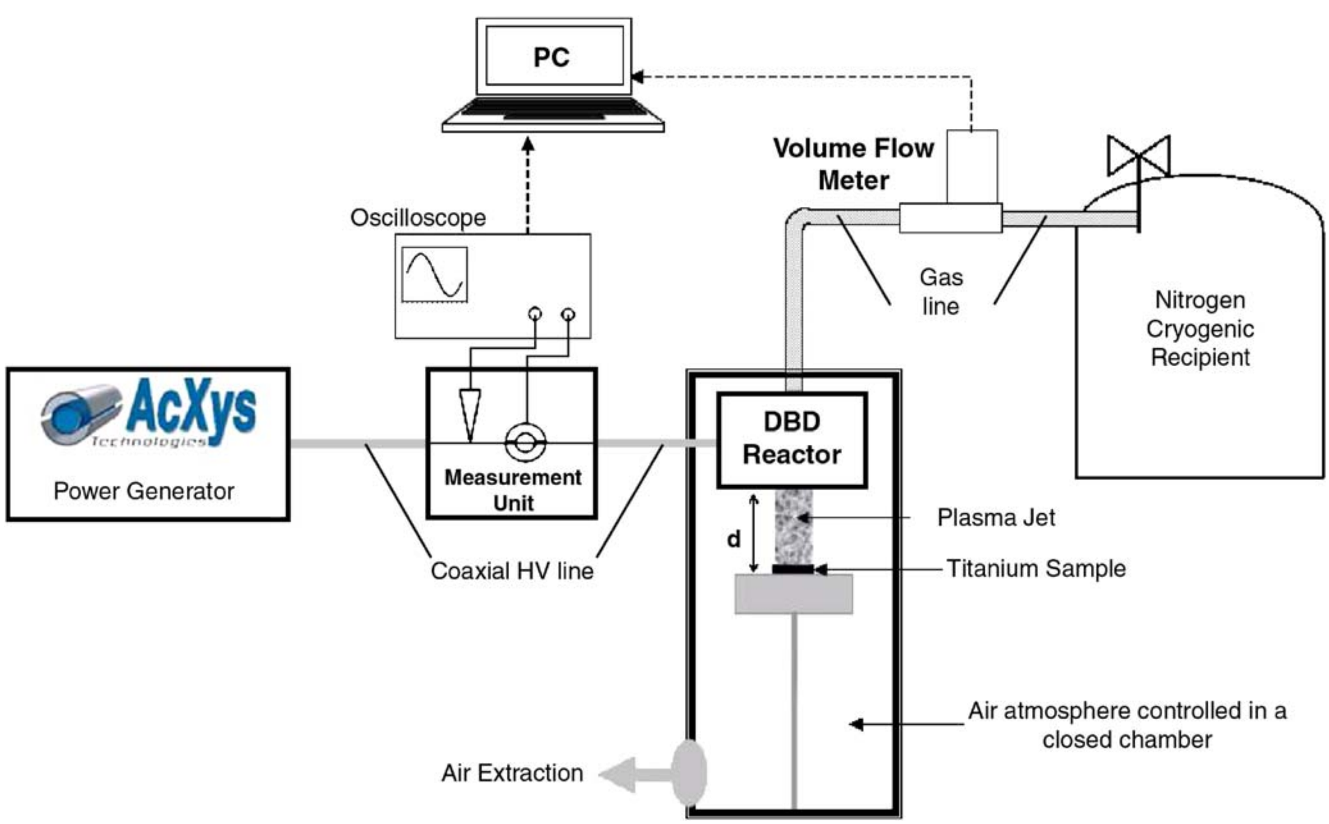

Fig. 1. Experimental set-up.