

## ALUMINIUM SURFACE ATMOSPHERIC PRESSURE DCSBD PLASMA TREATMENT IN AIR AND NITROGEN ATMOSPHERES

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**Abstract.** The results of Atomic Force Microscopy and Attenuated Total Reflectance FTIR for the analysis of aluminium surface changes by the Diffuse Coplanar Surface Barrier Discharge are presented in this contribution. Availability to use FTIR method for aluminium surface characterization was shown. Different behavior of surface changes was observed for different treating atmospheres: ambient air, nitrogen. Hypothesis about the possible reasons for different behavior was presented.

### 1. INTRODUCTION

Non-thermal cold plasmas working at atmospheric pressure are one of the most prospective tools for fast and cheap surface processing. Development of atmospheric pressure discharge physics allows implying plasma processing not only for high cost applications like microelectronics or space industry, but in-line industrial processes like production or processing of sheet or roll materials (for example see Tendero et al. 2006).

In this paper we present results about atmospheric pressure aluminium surface plasma processing using a special type of surface dielectric barrier discharge Diffuse Coplanar Surface Barrier Discharge (DCSBD). One nice example of aluminium sheets and foils industrial plasma processing is packaging industry which have total outcome in billions of euro. Past years the research of the atmospheric pressure plasma processing was intensely studied by different scientific groups. Infrared Spectroscopy in the case of our measurement on the surface of non-transparent materials Attenuated Total Reflectance Fourier Transform InfraRed Spectroscopy (ATR FTIR) was the method used for the current work. In scientific literature on ATR FTIR study on aluminium surfaces the method is mostly used for characterization of the polymer coated surfaces, like in work of Ohman et al. 2006. However we found that we can obtain nice results even on non coated aluminium surface. The results of such measurements are presented in this work together with AFM morphology study to see what changes the treatment atmosphere of the plasma on the aluminium surface properties are induced.

## 2. EXPERIMENTAL SAMPLES AND METHODS

Experimental samples in current work were produced by a DC magnetron sputtering of aluminium on glass substrates and thin aluminium sheets (material marking EN AW 1350) purchased normally in any metal distribution company. Prior to atmospheric pressure plasma treatment all samples were chemically precleaned using 5 min sonification in the mixture of isopropylalcohol and cyclohexane.

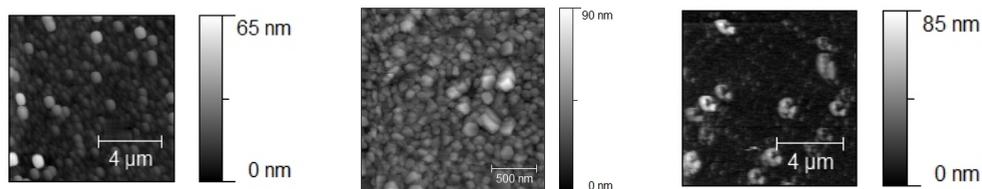
For the plasma treatment a novel atmospheric pressure Diffuse Coplanar Surface Barrier Discharge (DCSBD) was used (see Cernak et al. 2009). Plane coplanar electrode, surface and diffuse burning mode and possibility to start the discharge in different gas atmospheres allow using this plasma source with a working atmosphere as a control parameter of the surface treatment. The treatment conditions for aluminium surface were: i) the distance between coplanar electrode and sample surface 0.35 mm; ii) the power from a network 300 W (around 280 W to the discharge); iv) the treatment atmospheres were ambient air with relative humidity about 40% and nitrogen.

ThermoMicroscopes Autoprobe was used for the AFM measurements. The measurements were carried in ambient air using standard AFM tips in the contact mode.

Bruker VERTEX v80 spectrometer was used for Attenuated Total Reflectance FTIR (ATR FTIR) measurements. The spectrometer was equipped with MIRacle single reflection diamond ATR plate. The parameters for the measurements were: i) pressure in the chamber 25 Pa; ii) spectral range from  $600\text{ cm}^{-1}$  to  $4000\text{ cm}^{-1}$ ; iii) spectral resolution  $4\text{ cm}^{-1}$ ; iv) measurements were made as a result of averaging from 50 scans; v) spectra for each sample were measured as average from at least four different places.

## 3. PRELIMINARY RESULTS

AFM measurements of the surface topography showed that there was a large difference in surface changes for the DCSBD plasma treatment in ambient air and nitrogen atmosphere. Typical surface topographies of fresh, air and nitrogen atmospheric pressure DCSBD plasma treated DC magnetron sputtered aluminium surfaces are presented in Fig. 1. As can be seen the air DCSBD plasma induced great decrease of material grain size on the surface from about 700 nm to 100 nm, which is not observed in the case of the nitrogen DCSBD plasma treatment. We suppose that the reason for such different behavior is laid in presence of water vapors in air, which leads to an OH radical formation in the plasma. These radicals are the main specie in plasma making the changes on the aluminium surface. In the case of nitrogen atmosphere it looks like a partial etching occurred, because in our case we could not obtain the pure nitrogen in the discharge chamber.



**Figure 1:** AFM surface topography of DC magnetron sputtered aluminium surface: non-treated surface (left), 40 s air DCSBD plasma treatment (center), 40 s nitrogen atmosphere (right).

Attenuated Total Reflectance FTIR (ATR FTIR) was used to measure vibrations of chemical bonds presented on the surface. Usually in the case of metal surfaces this method is used when some polymer or other coating is deposited. However we found that aluminium surface due to its ability to create a native oxide layer in air atmosphere can be characterized by means of ATR FTIR. Reduced peaks amplitude allows us to conclude that practically we are gathering the ATR signal from the oxide layer.

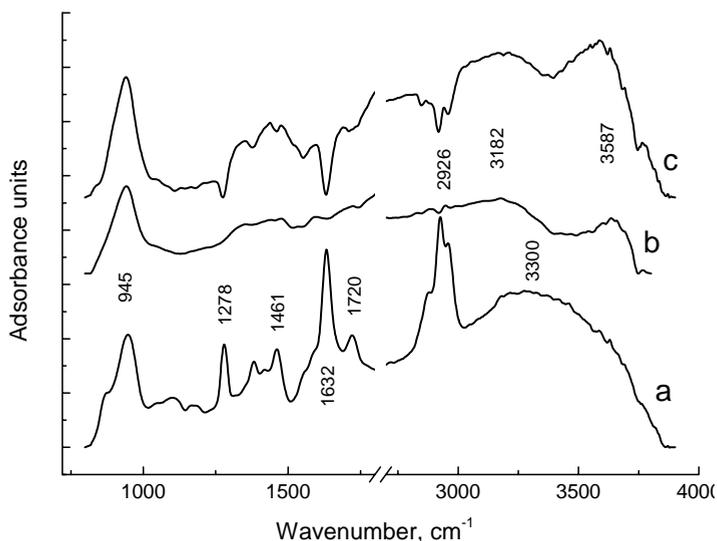
In the Fig. 2 is presented the typical view of ATR spectra of non treated aluminium surface (a), 40 s nitrogen DCSBD plasma treated surface (b) and 40 s air DCSBD plasma treated surface (c).

For the description of FTIR spectra mostly book of Mayo et al. 2004 was used. The peak with wavenumber  $945\text{ cm}^{-1}$  is corresponding to Al-O vibration and it is present in all measurements, showing the presence of an oxide layer.

The group of peaks around  $2900\text{ cm}^{-1}$  correspond to the C-H vibrations of  $\text{sp}^3$  and  $\text{sp}^2$  hybridized  $\text{CH}_x$ . And as it can be seen the nitrogen DCSBD treatment removes the peaks, but air plasma treatment giving peaks in the downside direction. This situation is due to the cleaning of the surface. It is so efficient that the surface contain less  $\text{CH}_x$  groups than background. The same behavior is observed for other types of C-H vibrations with wavenumbers  $1461\text{ cm}^{-1}$ ,  $1632\text{ cm}^{-1}$  and  $1720\text{ cm}^{-1}$ . Specific H-C-H wagging vibration is also presented on the non treated sample at wavenumber  $1278\text{ cm}^{-1}$  and it reveals the same evolution as C-H vibration peaks.

This can be one more prove that the main role of the surface cleaning in the case of DCSBD plasma is played by OH radicals. ATR FTIR spectra also contain the information about OH and water molecule vibrations and typically they located in the region  $3000\text{ cm}^{-1} - 3600\text{ cm}^{-1}$ . As it can be seen DCSBD plasma induces changes in the surfaces also in the OH and water containment in surface region. Wide peak with maximum at  $3300\text{ cm}^{-1}$  (in literature corresponding to water layer) is transforming into a group of two peaks with maximum at about  $3182\text{ cm}^{-1}$  (strongly bonded OH groups) and  $3587\text{ cm}^{-1}$  (weakly bonded OH groups).

Results obtained in this work allow us to find significant role of highly reactive OH radicals on aluminium surface cleaning and activation in DCSBD plasma. The source of OH radicals is in water vapors presented in ambient air.



**Figure 2:** Evolution of ATR FTIR spectra of aluminium surface: a) non treated aluminium surface; b) 40 s DCSBD nitrogen plasma treatment; c) 40 s DCSBD air plasma treatment.

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