Lateral force microscopy as a method of surface control after low-temperature plasma treatment

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Abstract. The surface properties of titanium, silicon, and a two-layer polymer film PMF-351 are studied after the action of a low-temperature plasma of dielectric barrier discharge (DBD). It is established that such treatment leads to the surface cleaning. This is evidenced by the values of roughness. The friction coefficient $C_f$ is selected as a characteristic that allows the time variation of surface properties after the plasma treatment. $C_f$ is determined using lateral forces regime of atomic force microscopy. The dependences of the friction coefficient $C_f$ on time for titanium, silicon, and a two-layer polymeric film PMF-351 are obtained. The best effect after treatment with low-temperature plasma persists for the first 20-30 minutes for titanium and a polymer film, and 5 minutes for silicon.

1. Introduction

Among the various methods of the surface modification without influence on the bulk properties of the material, the most promising one is the treatment in a low-temperature plasma of dielectric barrier discharge (DBD) [1,2]. Such plasma is used to improve the contact and adhesion properties of materials connection, in order to clean the contaminated surface. Due to the low temperature, this plasma has found application in medicine for sterilization, disinfection, and healing of living tissues. The ability to change the properties and the surface structure for a certain time is a unique property of this method. After that, the surface of the processed materials returns to the initial state.

There are various ways to control the properties and chemical structure of the materials processed in a low-temperature plasma [1]. Atomic force microscopy (AFM) is the most accurate method for estimating topography and surface properties (elastic modulus, adhesion, conductivity, and thermal conductivity) [3-8]. In addition to studying the surface properties of materials, AFM allows modifying the surface, carrying out tests for wear and friction [9-13], and exploring the time-varying processes. Changes in the surface properties after plasma treatment refer to such processes.

Often, modification by plasma does not lead to a change in the surface topography, determined by the high-resolution AFM method. However, other properties can change, for example, adhesion. An effective way of quantitative surface control can be measuring friction coefficient by AFM over time.

The aim of this work was to investigate the effects of low-temperature plasma treatment of a DBD on the surfaces of metal, polymer, and silicon using the method of the friction coefficient control of a surface over time.
2. Experimental details
To study the effect of a low-temperature plasma of a DBD on the surface properties, the following samples were chosen: silicon Si, metal Ti, and polymer 2-layer film PMF-351.

Samples were processed for 10 min by low-temperature plasma of DBD using a special setup (development of BSUIR) with working gas N₂, plasma source power about 10-30 W, and the distance from the source to the sample 20 mm.

The friction coefficient, adhesion, and microscale roughness of the samples were investigated using AFM device NT-206 (produced by MTM, Belarus) with the standard silicon probe type NSC-11 (MikroMash, Estonia) with the stiffness of the cantilever of 3 N/m. The surface roughness and friction coefficient ($C_f$) were investigated in scanning areas 20x20 µm².

The method of measuring the coatings friction coefficient using AFM is based on the estimation of the console twisting angle of the probe around its axis under the action of frictional forces between the surface and its tip [14]. The interaction of the AFM probe and the surface is also strongly affected by the adhesion forces between the probe and the surface; the larger the probe "sticks" to the surface, the greater the friction coefficient. After the plasma action, the adhesion forces increase and, consequently, the twisting of the console and $C_f$ increases.

3. Results and discussion
The microscale roughness $R_a$ is one of the criteria for the presence of unevenness and contamination on the surface.

According to the obtained results (Table 1), the surface microscale roughness $R_a$ for titanium and the polymer two-layer film PMF-351 film decreases twice after treatment with low-temperature plasma DBD. These results are in agreement with the data of works [1,2,15], which have established that plasma treatment cleans the surface. For smooth and pure silicon there are no significant changes in $R_a$ before and after treatment.

Table 1. Surface roughness of test samples before and after plasma treatment.

| Sample                  | Roughness before, nm (scanning area 20x20 µm²) | Roughness after, nm (scanning area 20x20 µm²) |
|-------------------------|-----------------------------------------------|----------------------------------------------|
|                         | $R_a$                          | $R_q$                          | $R_a$                          | $R_q$                          |
| Ti                      | 90.2                           | 106.2                          | 46.1                           | 59.5                           |
| Si                      | 5.8                            | 7.6                            | 4.8                            | 8.7                            |
| Two-layer film PMF-351  | 33.1                           | 41.6                           | 16.5                           | 20.7                           |

Surface treatment by the low-temperature plasma increases the two-layer film PMF-351 adhesion properties [1,15]. The friction coefficient between the surface and the probe tip is a quantitative parameter, depending on the adhesion forces. After treatment with the low-temperature plasma, the friction coefficient was fixed continuously for 75 min for the polymer film, 85 min for the titanium, and 20 min for the silicon.

Table 2 shows the mean values of the friction coefficient for initial samples without plasma treatment, maximum values after plasma treatment, values during 20 min after plasma treatment, and values during 75 min after plasma treatment.

Table 2. Mean values $C_f$ of tested samples before and after plasma treatment.

| Sample                | $C_f$ (initial samples) | $C_f$ (after treatment) | $C_f$ (20 min after treatment) | $C_f$ (75 min after treatment) |
|-----------------------|-------------------------|-------------------------|--------------------------------|--------------------------------|
| Ti                    | 0.21±0.001              | 0.23±0.01               | 0.140±0.007                    | 0.050±0.002                    |
| Si                    | 0.100±0.005             | 0.60±0.03               | 0.25±0.01                      | –                              |
| Polymer PMF-351       | 0.170±0.009             | 0.028±0.001             | 0.023±0.001                    | 0.015±0.001                    |
Figure 1. Time dependence of the friction coefficient for polymer PMF-351 after plasma treatment.

Figure 2. Time dependence of the friction coefficient for Ti after plasma treatment.

Figure 3. Time dependence of the friction coefficient for Si after plasma treatment.
The friction coefficient $C_{fr}$ remains constant at 0.05. The value 0.015 remains in the same range from 0.020 to 0.028 and during the next 35 min it decreases evenly to the final value 0.002.

Based on the results of the study, it is established that for all samples the friction coefficient decreases with time. For the two-layer film PMF-351, during the first 35 min after treatment $C_{fr}$ remains in the same range from 0.020 to 0.028 and during the next 35 min it decreases evenly to the final value 0.015 (Fig. 1). For titanium (Fig. 2), $C_{fr}$ decreases from 0.23 to 0.05 for the first 60 min, then remains constant at 0.05. For silicon (Fig. 3), $C_{fr}$ decreases from 0.6 to 0.25 through the entire period of time.

Adhesion of the titanium surface (Table 3) decreases with time after plasma treatment. This fact cannot be stated for a polymer film. The adhesion of the surface polymer film increases. The decrease in adhesion in titanium can be due to the cleaning of the surface from the oxide layers. It is assumed that treatment of the surface with low-temperature plasma leads to the change in the chemical structure of the surface layer of the polymer film [15] and leads to an increase in surface adhesion.

### Table 3. Adhesion force $F_{ad}$ and specific surface energy $\sigma$ of test samples before and after plasma treatment.

| Sample | Time after treatment, [min] | Adhesion forces $F_{ad}$, $\times 10^{-2}$ N | Specific surface energy $\sigma$, [N/m] |
|--------|-----------------------------|---------------------------------------------|----------------------------------------|
| PMF-351| 0                           | 0.79±0.04                                   | 0.039±0.002                            |
|        | 80                          | 1.57±0.07                                   | 0.078±0.004                            |
| Ti     | 0                           | 4.7±0.2                                     | 0.23±0.01                              |
|        | 80                          | 4.2±0.2                                     | 0.21±0.01                              |

The properties of the titanium surface, a two-layer polymeric film, and silicon after the treatment of low-temperature plasma of DBD were investigated by atomic force microscopy. It was established that the modified surfaces of various materials lost their active properties in different ways over the time. Processing in the plasma of the DBD cleaned the surface, which was confirmed by the roughness values.

The friction coefficient $C_{fr}$ of titanium immediately after treatment was practically unchanged compared to the initial one. $C_{fr}$ after 80 min has reduced by an order of magnitude.

Modified effect for 80 min better retained for the polymer film. $C_{fr}$ slowly declined over the entire period of time and decreased slightly.

The adhesion forces of the titanium surface after treatment reduced. The reason for the reduction was cleaning of the solid surface Ti from the oxide layers and pollutions, which provided adhesion. The adhesion forces of the polymer film surface after treatment increased due to the change in the chemical structure of the surface layer.

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