Deposition of fluoropolymer coating on stainless steel surface from plasma of glow discharge

A V Petrova\textsuperscript{1,2}, A L Bogoslovtseva\textsuperscript{1,2} and A I Safonov\textsuperscript{1}

\textsuperscript{1}Kutateladze Institute of Thermophysics SB RAS, Lavrentyev Ave. 1, 630090, Novosibirsk, Russia
\textsuperscript{2}Novosibirsk State University, Pirogova Str. 2, 630090, Novosibirsk, Russia

E-mail: safonov@itp.nsc.ru

Abstract. The present work investigates the application of the plasma of glow discharge to deposit the fluoropolymer coatings. Two series of experiments were carried out in which the influence of pressure and current strength on surface coatings morphology during its surface treatment with glow discharge plasma was investigated. The surface morphology is investigated by a scanning electron microscope (SEM), and the water contact angle (WCA) is measured. The fact that the choice of pressure significantly affects the morphology and properties of the deposited coating is established.

1. Introduction
Surface functionalization is one of the main technologies used in modern technology. The microstructure and nanostructure elements on the material surface can cause the appearance of the required properties, for example, wettability [1-4]. Glow discharge is one of the simple and common methods for cleaning and pretreating surfaces before the coating. Generally, either argon or atmospheric air is used as a working gas. But it is precisely the possibility of using a glow discharge to activate a precursor gas of fluoropolymer coatings that is of great interest. Hydrophobic and superhydrophobic fluoropolymer coatings with high adhesion can be deposited as the result.

The main goal of the presented work was to investigate influence of parameters of glow discharge on the activation of precursor gas. Precursor gas pressure and current of glow discharge during the treatment process were used as investigate parameters.

2. Experimental details

2.1. Experimental setup
The scheme of the experimental setup is shown in Figure 1. The experimental setup consists of: a vacuum chamber with a volume of 30 liters; vacuum pumping systems based on an oil-free vacuum pump; systems for measuring and monitoring gas-dynamic parameters (vacuum gauge); precursor gas supply systems with flow mass controllers (FMC); high-voltage power source; electrodes and a system of high-voltage leads to the chamber. Two electrodes with a diameter of 20 mm are placed in a vacuum chamber opposite each other. The distance between the electrodes is 15 mm. Both electrodes are insulated from the chamber walls and connected via wires to a high-voltage DC power supply. The source VIP-7.5-0.3 is capable of delivering voltage up to 3 kV and current up to 150 mA. The regulated parameter in the source is the current. The discharge voltage and current are monitored using...
a voltmeter and an milliammeter, respectively, installed in a high-voltage direct current source. Hexafluoropropylene oxide (C₃F₆O) was used as the precursor gas of the fluoropolymer coatings. The stainless-steel (AISI 321) discs with a diameter of 20 mm and a thickness of 2 mm were used as the samples.

![Scheme of the experimental facility](image)

**Figure 1.** Scheme of the experimental facility.

There were two series of experiments to get more knowledge about the deposition of fluoropolymer coatings. First, the investigated parameter was pressure. It takes values from 0.1 to 5 Torr. Other deposition parameters were constant and equal $U = 700$ V, $I = 100$ mA, $t = 20$ minutes, $Q = 25$ sccm. In the second series of the experiments, the current in process is changed and takes values from 10 to 100 mA. Other deposition parameters were constant and equal $U = 750$ V, $P = 0.5$ Torr, $t = 20$ minutes, $Q = 25$ sccm.

### 2.2. Diagnostic methods

The surface morphology of the obtained samples is analyzed using the JEOL JSM6700F scanning electron microscope. The water contact angle (WCA) was measured using a DSA-100E device, KRÜSS GmbH.

### 3. Result and discussion

The surface morphology after deposition is shown in Figure 2. At low pressures (of the order of 0.1 Torr, Figure 2a), dendritic structures are formed on the previously cleaned surface of the sample, covering the surface. Water contact angle (WCA) measurements prove the formation of hydrophilic coatings (Figure 2a). This value of WCA is very close to the value on the surface of stainless steel. This may indicate that the resulting coatings are thin and uneven. With increasing pressure, the morphology of the coating changes: at a pressure of 0.5 Torr, the coating is spherical structures with a size of 200 - 700 nm (Figure 2b). WCA measurements prove the formation of hydrophobic coatings. WCA reaches 145°. This value is significantly higher than for smooth the Teflon. This indicates that wetting is influenced by the nanostructure of the surface, which confirms the results of surface morphology. A further increase in the pressure from 1 to 5 Torr entails the formation of layers of continuous coatings (Figure 2c, d). Obtained hydrophobic coatings are formed with the WCA values that are typical for fluoropolymer coatings: the values change from 115° to 125° the surface.
morphology of the samples when applying treatment with different values of current strength is shown in Figure 2.

![Figure 2. SEM images of the sample surface versus pressure.](image)

Figure 3 shows the images of the surface of the samples after treatment by glow discharge plasma with different current. It can be noted that with an increase in the current strength, the structure of the coating changes from smooth (at 10 mA, Figure 3b) to globular at a current strength above 25 mA (Figure 3c). At a current of about 50 mA (Figure 3d), globules tend to take the shape of a sphere with an average size of about 150-300 nm. Further, with an increase in the current, the number of globules significantly increases (Figure 3e, f).

Measurements of the water contact angle depending on the specified current strength are shown in Figure 3 (inserts). It can be noted that with the used processing parameters, a hydrophobic coating is formed even at a current of 10 mA. The maximum WCA value was reached at a current of 50 mA, and a superhydrophobic coating was formed (WCA is more than 150°). The dependence of the WCA on the current strength, confirms the assumption that the formation of a coating is homogeneous in composition.
Figure 3. SEM images of the sample surface depending on the value of the discharge current.

Summary

Fluoropolymer coatings with hydrophobic and superhydrophobic properties were deposited on the stainless-steel samples by using glow discharge plasma.

The choice of pressure significantly affects the morphology of the deposited coating. In the process of increasing pressure, one can observe the process of changing the morphology from dendrite-like structures to continuous coatings including the step of the coatings consisting of spherical particles.

The value of the current of glow discharge allows to control the property of surface wetting. Choosing a current strength above 25 mA leads to the productive formation of coatings with a high fluorine content, which has the property of high hydrophobicity to a large extent.

Acknowledgement

This work is executed from a state contract with IT SB RAS (121031800218-5).

References

[1] Drelich J and Marmur A 2014 Surface Innovations 2 211
[2] Surtaev A S, Serdyukov V S and Pavlenko A N 2016 Nanotechnologies in Russia 11 696
[3] Boinovich L B and Emelyanenko A M 2008 Russ. Chem. Rev. 77 583
[4] Safonov A, Sulyaeva V, Gatapova E, Starinskiy S, Timoshenko N and Kabov O 2018 Thin Solid Films 653 165