Structure of microplasma discharge on titanium surface covered with thin dielectric film

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Abstract. The spatial structure of the pulsed microplasma discharge (discharge duration is 100 μs, the amplitude of the discharge current is 200 A) was experimentally studied. The discharge is initiated on the titanium sample surface covered with the thin dielectric (titanium oxide) film with a thickness of ~10 nm. The sample was exposed to the pulsed plasma flow (the plasma density is $2 \times 10^{13}$ cm$^{-3}$, and the pulse duration is 25 μs). It was found that the microplasma discharge glow has the branched dendrite structure, which consists of a large number of cathode spots (brightly glowing localized formations) randomly distributed over the metal surface. As a result of interaction between the microplasma discharge and the titanium sample, erosion occurs on its surface. The erosion structure is visually “identical” to the discharge glow structure. It was ascertained that the erosion pattern on the titanium surface consists of a large number of individual microcraters with characteristic sizes from 0.3 to 10 μm randomly distributed over an area of approximately 1 cm$^2$. All together, these microcraters form the branched dendrite structure. It was also ascertained that the microplasma discharge propagates along the titanium surface covered with a thin dielectric (titanium oxide) film at an average velocity of approximately 70 m/s, and this propagation has a “jumping” character: the plasma of burning cathode spots initiates the excitation of new cathode spots at distances of 3–30 microns from them.

1. Introduction

It is well known that, in vacuum, the pulsed plasma flow (with a density of more than $\sim 10^{11}$ cm$^{-3}$ and duration of several microseconds) can initiate the microplasma discharge on the metal surface covered with thin dielectric (metal oxide) films with thicknesses of $d \sim 1$ μm [1–3]. For the stable initiation of these discharges, it is important that the metals should have the negative electric potential of 100–400 V, and the dielectric film should be damaged at least in one region on the open metal surface [1]. In this case, the plasma pulsed flow interacts simultaneously with the dielectric film and the open metal surface, and the outer surface of the dielectric film becomes charged due to the plasma ion flow and acquires the floating electric potential $\phi_f$ (as a rule, the floating potential is close to the potential of the grounded vacuum chamber wall, i.e. $\phi_f \approx 0$). But the metal sample has a negative electric potential of $\phi_m = -400$ V (before the discharge initiation). Under these conditions, the electric field between the outer surface of the dielectric film and the open surface of the metal is as follows [1]:

$$E = |\phi_m - \phi_f|/d \approx |\phi_m|/d$$

In our previous experiments on microplasma discharge excitation on the titanium surface by the pulsed plasma flow, as a rule, we used the titanium samples covered with the 1-μm-thick dielectric oxide films. To obtain such films, the titanium samples were heated in air at the atmospheric pressure...
at a temperature of 400°C. In this case, when the pulsed plasma flow falls onto the titanium sample covered with the dielectric film, the electric field of approximately 4 MV/cm arises between the outer surface of the dielectric film and the inner film surface, which is in contact with the metal titanium surface [4]. This electric field is strong enough to initiate the electrical breakdown across the dielectric film cut in those areas where the film is damaged and the cut is near the open metal surface. Due to the electrical breakdown, the dense plasma of local cathode spots is formed on the open metal surface. The special power supply maintains the 200 A electric current of microplasma discharge, which results in the film destruction and the formation of microcraters in the local molten regions on the metal surface [5, 6]. In the stage of microplasma discharge burning, the potential of the metal decreases to 30–50 V. The probability of the microplasma discharge excitation will be approximately 90%, if the oxide film thickness on the titanium surface is $d \sim 1 \, \mu m$ [5, 6].

For the titanium samples covered with the oxide films, which are thicker or thinner than 1 μm, the probability of the microplasma discharge excitation on their surface is less [5, 6]. Indeed, it can be seen from the above formula (1) that, if the film thickness increases from 1 to 10 μm, then the electric field at the film cut will decrease by an order of magnitude, and, therefore, the probability of the microplasma discharge excitation by the pulsed plasma flow will be also considerably reduced. As the film thickness on the titanium surface decreases from 1 to 0.01 μm, its electrical conductivity considerably increases [7, 8], and the electric charge density on its surface becomes considerably less, since the ion flow from plasma with a density of $\sim 10^{11} \, \text{cm}^{-3}$ cannot charge the outer film surface up to the plasma floating potential. Therefore, the electric field between the outer film surface and the metal is considerably reduced, and the probability of microplasma discharge excitation is reduced to approximately 1% [5, 6]. Similar results on the efficiency of microplasma discharge initiation on the metal surfaces were also obtained for the aluminum and steel samples that confirm that this efficiency depends on the dielectric film thickness [3, 9, 10].

Thus, at the optimal dielectric film thickness of 1 μm on the titanium, steel, and aluminum samples, the microplasma discharge is always initiated at the boundary between the dielectric film cut and the open metal surface. The velocity of microplasma discharge propagation along the titanium surface is approximately 1 m/s. It is determined by the process of the dielectric film evaporation under the effect of the dense plasma of microdischarges (cathode spots). After the metal sample covered with 1-μm-thick dielectric oxide film is 10 times exposed to the microplasma discharge with duration of 20 ms, the film on its surface becomes completely destroyed, and the pronounced microrelief with a characteristic roughness of 1–2 μm is formed on the metal surface [9–12]. Such new method for forming the long-living microrelief on the metal surface using microplasma discharges is in demand in medicine, including the production of the orthopedic and dental prosthesis, as well as in the industrial production of composite materials.

However, the spatial structure is not yet studied of the microplasma discharge initiated on the titanium surface covered with a continuous thin dielectric oxide film with a thickness of $d \approx 2–10 \, \text{nm}$. It is known from the scientific publications that, in air at room temperature, it takes several minutes to form such a thin film on the titanium surface precleaned in vacuum, which forms as a result of the metal titanium oxidation by atmospheric oxygen [13]. After the sample stays in air for a long time (for many hundreds of hours), the oxide film thus formed on the titanium surface turns out to be very stable, and its thickness almost does not increase, being of the order of 10 nm [14].

It is important to note that this thin dielectric oxide film is continuous, without gaps and cuts, and covers the entire metal sample. Thus, when the external pulsed plasma flow interacts with such a sample, there is no contact between the plasma and the open metal surface. In this case, the electric field arising between the outer oxide film surface and the metal is concentrated inside the film, and therefore, the electrical breakdown across the dielectric film cut is impossible. Thus, in order to initiate the microplasma discharge on the titanium surface covered with the 10-nm-thick oxide film, the local electric field should be strong enough to initiate the electrical breakdown in the bulk of the continuous dielectric film.

In the experiments presented, the spatial structure is studied of the glow of a single pulsed microplasma discharge initiated by the dense pulsed plasma flow on the titanium surface covered with
a continuous ultrathin dielectric oxide film with a thickness of 10 nm. In this case, it was also interesting to study the structure of the erosion pattern arising on the titanium surface as a result of excitation of a single microplasma discharge.

2. Experiment
The studies were carried out at the “Sphere” experimental device [1–6, 9, 15], which is the spherical stainless steel vacuum chamber with a diameter of 50 cm, pumped out to a residual air pressure of \(~1\) Pa (Figs. 1a, 1b). A cylindrical-type plasma injector was installed on the side flange of the vacuum chamber, in which the pulsed plasma flow was formed as a result of the high-voltage electrical breakdown (7 kV, 1.5 kA, pulse duration is 15 \(\mu\)s) on the organic glass surface. The pulsed plasma flow comes into the chamber. The main characteristics of the pulsed plasma flow, such as the electron temperature, the charged particle density, and the pulse duration, as well as their space-time variations were measured using the Langmuir probe. As the plasma flow propagates from the injector output to the chamber center, it expands and its density decreases from \(2 \times 10^{13}\) cm\(^{-3}\) (at a distance of 2 cm from the injector output) to \(5 \times 10^{10}\) cm\(^{-3}\) (in the center of the chamber). The electron temperature at the plasma pulse leading edge (1–5 \(\mu\)s) was approximately 10 eV at any distance from the injector, and it remained almost unchanged as the plasma expanded.

![Figure 1a. Experimental vacuum chamber “Sphere” for studying microplasma discharges excited on the metal samples.](image)

![Figure 1b. Schematic of the experiment:](image)

In the experiment, the plane titanium sample with sizes of \(20 \times 20 \times 1\) mm\(^3\) was used. This plate was covered with the continuous dielectric oxide film with a thickness of \(~10\) nm, which formed naturally on the sample surfaces when it was kept in air at room temperature for several hours [16]. Since the 10-nm-thick dielectric film has the considerable electric conduction [7, 8], the probability of the microplasma discharge excitation on the titanium sample covered with such a film and exposed to the pulsed plasma flow with the low density of \(~10^{11}\) cm\(^{-3}\) is less than 1\% [5, 6]. Therefore, for the stable electric charge accumulation on the outer surface of the film (and, accordingly, for
the reliable excitation of the microplasma discharge on the titanium surface), it was necessary to use the pulsed plasma flow with the considerably higher density of $2 \times 10^{13}$ cm$^{-3}$.

The sample was installed at a distance of approximately 2 cm from the plasma injector output. The leading edge of the plasma pulse (with a plasma density of $2 \times 10^{13}$ cm$^{-3}$) reached the sample in five microseconds after the beginning of the plasma flow generation (Fig. 2).

Exposing the titanium sample surface to a single plasma pulse, it was possible to excite the microplasma discharge, which was characterized by the burning voltage of approximately 50 V, the total electric current of 200 A and the pulse duration of 100 μs. Waveforms of the voltage and current in the microplasma discharge are presented in Fig. 3.

Using the Nikon D7100 camera, the glow was recorded of a single microplasma discharge excited on the titanium sample surface (Fig. 4). Since the camera operated with the opened shutter, the exposure time was determined by the duration of the microplasma discharge glow, which was approximately 100 μs and, in turn, was determined by the duration of the electric current pulse.

Figure 4 shows that the structure of a microplasma discharge glow on the titanium surface covered with a 10-nm-thick oxide film is strongly inhomogeneous and has a characteristic tree-like branching shape (dendrite shape). To reveal the fine structure of the glow, the dendrite-shaped image was further
processed, namely, it was converted to a black-and-white image, the image contrast was increased, and the halo glow of the plasma surrounding the sample was removed. As a result, it was found that the microplasma discharge glow consists of many localized glowing objects with characteristic sizes of 100–200 μm, located inside the dendrite region. The results of processing such dendrite image are presented in Fig. 5. Figures 4 and 5 demonstrate that the microplasma discharge glow is produced by a large number of localized glowing objects. These objects are almost motionless during the exposure time of 100 μs and have a round shape. If the microdischarges moved rapidly, the round shape of their glow would be transformed into the ellipse-like shape, but this is not seen in the images. Therefore, we can assume that the maximum displacements of microdischarges (cathode spots) do not exceed 10% of their characteristic diameters (which are 100–200 μm), i.e. they are less than 10–20 μm. So, we can estimate that the velocity of individual cathode spot averaged over 100 μs is not more than 10–20 cm/s.

Figure 4. Image of the glow of a single microplasma discharge excited on the titanium sample surface covered with a natural dielectric oxide 10-nm-thick film. The external pulsed plasma flow with a density of $2 \times 10^{13}$ cm$^{-3}$ falls onto the sample from the right, where the plasma injector was installed. The titanium plate with dimensions of $20 \times 20 \times 1$ mm$^3$ is installed so that its edge faces the incident plasma flow. The real dimensions of the glowing dendrite-shaped discharge region are $7 \times 8.5$ mm$^2$.

Figure 5. High-contrast image of the microplasma discharge glow taken with an exposure time of 100 μs. The real dimensions of the glowing region are $7 \times 8.5$ mm$^2$. The microplasma discharge was excited on the surface of a titanium sample (with the 10-nm-thick dielectric oxide film) by the pulsed plasma flow falling onto it from the right. This image was obtained as a result of converting the colored image into the black-and-white one, and removing the plasma glow halo.

On the other hand, it is known that the microplasma discharge always occurs in those regions of the sample that are exposed to the denser plasma flow, i.e. are closer to the plasma injector. Indeed, in our case, the microplasma discharge was initiated by the pulsed plasma flow on that edge of the sample, which is the closest to the plasma injector. After this, the microplasma discharge propagated from the sample edge to its center, i.e., from right to left (Figs. 4, 5). Since the discharge glow region with dimensions of approximately $7 \times 8.5$ mm$^2$ was formed during 100 μs, the average velocity of the microplasma discharge propagation along the titanium surface is 70 m/s.
Thus, we found the strong contradictions in the results of estimating the propagation velocities of microplasma discharge along the titanium surface covered with the dielectric film. For the titanium surfaces covered with the thick (1 μm) and thin (10 nm) dielectric films, the propagation velocities are 1 and 70 m/s, respectively, and in the latter case, the local microdischarges (cathode spots) are almost motionless. In order to explain such strong contradictions, the local erosion on the titanium sample surface covered with the 10-nm-thick dielectric film was studied. Macro-scale photographs of the erosion zone on the titanium plate produced as a result of its exposure to one microplasma discharge are presented in 6.

![Figure 6a](image1.png)

**Figure 6a.** Macro-scale image of erosion zone produced on the titanium sample surface covered with 10-nm-thick oxide film as a result of excitation of a single microplasma discharge with duration of 100 μs and an electric current amplitude of 200 A. The sizes of the erosion zone are 7 × 8.5 mm².

![Figure 6b](image2.png)

**Figure 6b.** The same image as in **Figure 6a** after the inversion of colors and increasing the image contrast performed to improve image quality of the erosion zone local structure. The sizes of the erosion zone are 7 × 8.5 mm².

3. **Discussion**

Based on the comparative analysis of the images presented in Figs. 4, 5, 6, we can conclude that, at the same enlargement of the images, the structures of the surface erosion and the microplasma discharge glow are visually almost “identical”, and arise as a result of the microplasma discharge excitation on the titanium surface. The detailed erosion structure is shown in the micrograph of the erosion zone fragment on the titanium sample surface, which appears as a result of the excitation of one microplasma discharge with an electric current amplitude of 200 A and pulse duration of 100 μs (Fig. 7). This micrograph was obtained using the scanning electron microscope with a magnification of ×800. Figure 7 shows that the erosion zone on the titanium surface consists of a large number of individual microcraters with characteristic sizes of 0.3–10 μm randomly distributed over the sample surface, and their locations correspond to the brightly glowing “points” of the microplasma discharge. These microcraters arise on the sample as a result of the local melting of titanium, which is characteristic of the process of the explosive electron emission on the metal surface in the strong local electric field [17–21]. The distance between the individual microcraters varies from 3 to 30 microns.
It is important to note that we do not observe the traces of erosion on the sample, which have the forms of “continuous” chains of microcraters or lengthy molten tracks (channels). This indicates that the microdischarges initiated in some local points (cathode spots) do not move over the metal surface, but burn in these points for a short time, and then disappear. During the primary microdischarge burning, the plasma produced by them initiates the secondary microdischarges in the neighboring local regions (at distances of 3–30 μm from the primary microdischarges). In this case, the number of microdischarges and microcraters on the titanium sample surface per unit area of the erosion zone reaches $10^5$–$10^6$ cm$^{-2}$ (Fig. 7). Thus, these regular cyclic processes of occurrence, burning, and subsequent quenching of microdischarges (cathode spots) on the titanium surface are repeated many times. Since a current of approximately 10 A is necessary for a single cathode spot burning [22], it can be assumed that, at the total discharge current of 200 A, approximately 20 cathode spots are burning simultaneously. Thus, in order to $10^5$–$10^6$ erosion craters to arise, it is necessary that at least $10^4$–$10^5$ cycles should occur during the pulsed microplasma discharge with duration of 100 μs.

It is important to note that, though the duration of the external pulsed plasma flow is 25 μs, the microplasma discharge on a titanium surface covered with 10-nm-thick oxide film is initiated by the plasma with the maximum density of $2\times10^{13}$ cm$^{-3}$ which exists only at the leading edge of the plasma pulse (Fig. 2), so, the microplasma discharge is initiated only on the edge of the titanium sample during the first 2 μs, and all the subsequent localized microdischarges with the total duration of 98 μs are excited not by the external pulsed plasma flow, but by the dense plasma of the primary and subsequent microdischarges, which are a part of the repeated cycles of excitation, burning, and quenching of new microdischarges throughout the entire 100-μs-pulse. During this process, new microdischarges are excited and localized at distances of 3–30 μm from the previous discharges, and their dense plasma initiates the next microdischarges. Thus, the microplasma discharge propagation over the titanium surface covered with the thin 10-nm-thick oxide film occurs by means of “jumping” from one cathode spot to another at an average velocity of approximately 70 m/s: Indeed, after their excitation, the individual microdischarges do not move over the surface, but they generate and inject the dense plasma, which charges the oxide film on the titanium surface and initiates new microdischarges in the vicinity of the already burning ones.

Where do new localized microdischarges occur? It is obvious that the dense plasma of burning microdischarge most strongly affects the sample surface in the neighboring regions, since the strong electric field arises between the dielectric film and metal. Therefore, new microdischarges are the most likely to be initiated in the immediate vicinity of already burning microdischarges, at distances of 3–30 microns from them. Let us more exactly determine the points of their initiation.

Figure 7. Microphotograph of the erosion zone fragment on the titanium sample surface. The erosion occurred as a result of the sample exposure to a single microplasma discharge with the electric current amplitude of 200 A and pulse duration of 100 μs. The micrograph was obtained using the scanning electron microscope with a voltage of 20 kV at the cathode of the electron gun. The horizontal image size is 104 μm and the vertical size is 112 μm.
The dielectric oxide film formed in air on the titanium surface is inhomogeneous [13, 14], and its thickness varies from $d_1 = 2$ nm to $d_2 = 10$ nm. Taking into account the fact that, during the discharge burning, the voltage on the metal decreases to $\Phi_m \approx -50$ V (Fig. 2), for two film thicknesses of $d_1 = 2$ nm and $d_2 = 10$ nm and for $\Phi_f \approx 0$, formula (1) gives the following electric field strengths: $E_1 \approx |\Phi_m|/d_1 = 250$ MV/cm and $E_2 \approx |\Phi_m|/d_2 = 50$ MV/cm, respectively. These electric field strengths are both quite sufficient for the occurrence of the explosive electron emission from the metal surface accompanied by the crater formation [23–26].

However, it is known [7, 8] that the thinner oxide films (with $d_1 = 2$ nm) have the considerably higher electrical conduction, which increases disproportionately to a decrease in the film thickness. On the one hand, even in the dense plasma flow, this prevents the surfaces of the ultrathin oxide films from the charge accumulation on them, and, thereby, can considerably reduce both the electric field on its surface and the probability of new microplasma discharge initiation by the plasma of burning microdischarges. On the other hand, for the thicker oxide films (with $d_2 = 10$ nm), the electrical conduction is much lower, therefore, on the surface of such a film, the electric charge is accumulated more efficiently and reaches the stationary value faster, and its potential becomes equal to the plasma floating potential. Taking these considerations into account, we can conclude that, in reality, the electric field $E_2$ may be larger than the field $E_1$, and new microdischarges will be more likely initiated on the titanium surface in those regions where the film is thicker.

4. Conclusions

The glow spatial structure was experimentally studied of the pulsed microplasma discharge initiated on the titanium sample surface covered with the ultrathin continuous dielectric oxide film with a thickness of 2–10 nm. The microplasma discharge was initiated on the surface of the titanium plate ($20 \times 20 \times 1$ mm$^3$) by the external pulsed plasma flow (density is $2 \times 10^{13}$ cm$^{-3}$, pulse duration is 25 μs). Subsequently, the plasma of the primary microdischarges (cathode spots) initiated the secondary and the next microdischarges. Such repeated cycles of occurrence, burning, and generation of new microdischarges were supported by a total current of 200 A at the pulse duration of 100 μs.

It was ascertained that the glow structure of the microplasma discharge has the form of a branched dendrite, which consists of a large number of brightly glowing localized microdischarges.

As a result of the microplasma discharge effect on the sample, the erosion occurs on its surface, the structure of which is visually “identical” to the discharge glow structure. It consists of a set of individual microcraters with characteristic sizes from 0.3 μm to 10 μm covering an area of approximately 1 cm$^2$. These microcraters are produced in those local points, where numerous microdischarges are initiated, burning for some time and then quenched. During the process of microdischarge burning on the titanium surface, the plasma of primary microdischarges initiates the secondary and the next microdischarges in the neighboring regions at distances of 3–30 μm from the primary microdischarges. Thus, these cyclic processes of the initiation of new and quenching of the “old” microdischarges are repeated many times: not less than $10^2$–$10^5$ cycles occur during 100 μs.

It has been experimentally shown that the single microplasma discharge propagates along the titanium surface covered with the thin dielectric film with a thickness of ~10 nm at an average velocity of 70 m/s. On a microscale, the microplasma discharge propagates by means of numerous “jumps” from primary local cathode spots to the next ones (initiated by the previous burning cathode spots), leaving a “trace” consisting of many separate microcraters, which all together look like a branched dendrite.

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