

## PACKET-PULSE DUAL MAGNETRON SPUTTERING

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*The paper presents the results of experimental study of the discharge formed by a dual (DU) magnetron sputtering system (MSS) with aluminum targets in the mode of packet-pulse magnetron sputtering of high power (called deep oscillation magnetron sputtering (DOMS) in foreign literature). A special feature of the discharge in the DOMS mode is the use of unipolar sequence of micropulses with short durations and high powers that form macropulse duration 1000–3000  $\mu$ s. This mode of sputtering was previously used only in single MSS. In this paper, the DOMS mode is first investigated with a dual magnetron sputtering system. The main plasma parameters are measured using triple and single Langmuir probes. Dependences of plasma parameters on the parameters of the pulsed discharge power supply: voltage and current amplitudes, current density, and power density on the target surface are established. The results of experiments show that the use of the dual packet-pulse magnetron sputtering can significantly increase the plasma density and the ion current density on the substrate in comparison with the traditional DC and MF modes of magnetron sputtering. The ratio of the ion flux density to the flux density of neutral atoms, characterizing the degree of ion bombardment of the growing coating in the DU DOMS mode, reached a value of 28, whereas in the DC mode it was 0.8.*

**Keywords:** DOMS, HIPIMS, dual magnetron sputtering system, reactive magnetron sputtering.

### INTRODUCTION

Methods of ionized physical vapor deposition of coatings, such as high power impulse magnetron sputtering (HIPIMS) [1–3] and modulated pulse power magnetron sputtering (MPPMS) have actively been developed recently [4, 5]. Both methods provide high plasma density and ionization degree of sputtered material, which allows coatings to be fabricated with characteristics that can hardly be reached by the direct current magnetron sputtering. A modification of the MPPMS method is the packet-pulse magnetron sputtering of high power called deep oscillation magnetron sputtering (DOMS) in the English-language literature [6–8]. The essence of the method is that unipolar voltage pulses with duration of 1000–3000  $\mu$ s are applied to the discharge gap of the magnetron sputtering system (MSS). Each macropulse consists of a packet of micropulses with durations of 3–20  $\mu$ s and pulse repetition frequency of 5–60 kHz. The repetition frequency of pulses in the packet lies in the range from 50 to 300 Hz. This relatively new technology has great prospects for solving problems in which the ionization degree of the deposited material and the degree of ion bombardment of the growing coating are important. In addition, the results published over the past few years showed that the use of the DOMS method for dielectric coating deposition allows reactive magnetron sputtering to be performed practically without arc formation [9].

In the present work, the results of study of the discharge formed by a dual MSS operating in the bipolar high power deep oscillation magnetron sputtering mode are presented. The use of the dual MSS allows the plasma density and the degree of sputtered target material ionization in the region of the substrate to be increased in comparison with

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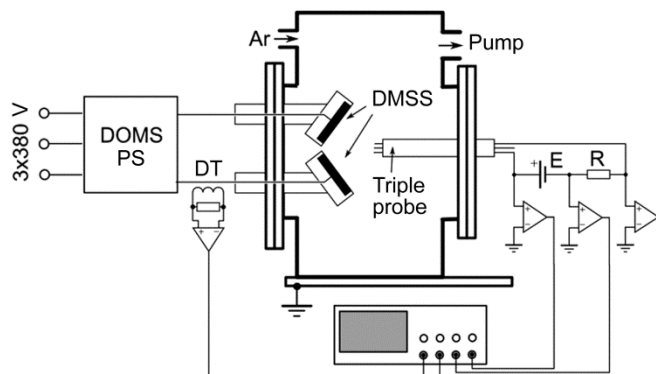


Fig. 1. Schematic of the experimental setup for a study of the discharge formed by the DU DOMS system.

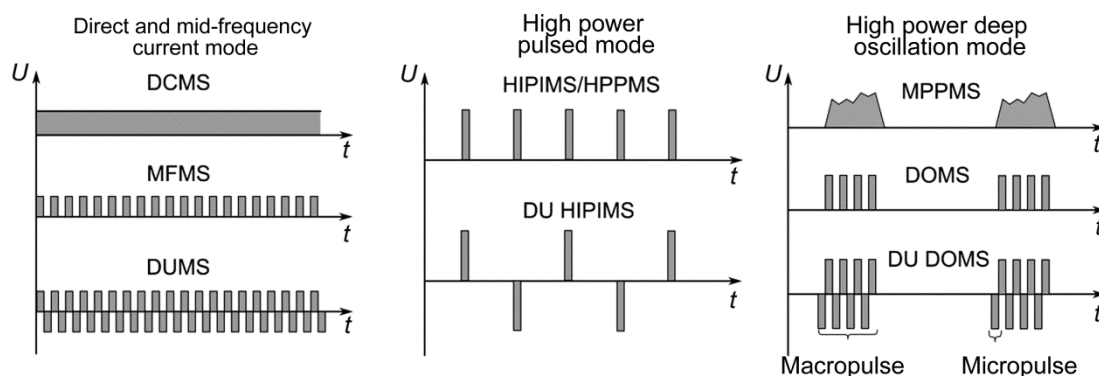


Fig. 2. Voltage plots for the indicated magnetron sputtering modes.

a single system. As a result of alternating operation of magnetrons during the macropulse, a high degree of ion bombardment of the growing coating is continuously maintained.

## EXPERIMENTAL EQUIPMENT

To study the DU DOMS discharge, an experimental setup the schematic of which is shown in Fig. 1 was used. A 215-liter vacuum chamber was pumped out with a turbomolecular pump to a residual pressure of  $6 \cdot 10^{-3}$  Pa. A dual magnetron sputtering system (DMSS) with plane round aluminum cathodes having a diameter of 76 mm and a thickness of 5 mm was placed at the side wall of the chamber. The DMSS cathodes with direct water cooling were used. The power of the sputtering system in the continuous mode can reach 3 kW (power density on the target surface was  $0.03 \text{ kW/cm}^2$ ). The magnetic system of the magnetrons formed by the inner cylindrical and outer ring NdFeB magnets was slightly imbalanced with the geometric imbalance coefficient  $K_g = 1.2$  [10]. The magnetic field induction on the surface of the target cathode in the *magnetic arc* zone was 730 G. The DMSS had a closed configuration of the magnetic field. The angle between the magnetron target surfaces was  $158^\circ$ .

To power the DMSS, a special power supply (PS) capable of operating not only in the conventional, but also in high power impulse magnetron sputtering mode was developed. Figure 2 shows voltage plots of pulses generated by the PS in various operating modes. In the present work, the following modes were used: direct current magnetron sputtering (DCMS) mode, dual mid-frequency pulsed magnetron sputtering (DUMS) mode, and high power deep oscillation

TABLE 1. DOMS PS Parameters

Output voltage	100–1500 V
Average output current	up to 12 A
Average output power	up to 10 kW
Maximum pulse current	200 A
Maximum pulse power	300 kW
Macropulse frequency	1–1000 Hz
Macropulse duration	100–3000 $\mu$ s
Micropulse frequency	10–50 kHz
Micropulse duration	3–50 $\mu$ s
Stabilization regimes	voltage/current/power
Operation regimes	DCMS/DUMS/DU-DOMS

magnetron sputtering (DU DOMS) mode. The device and the operating principle of the developed power supply are described in [11, 12]. Its main parameters are presented in Table 1.

A special feature of the PS is an increased level of the output voltage ( $\pm 1500$  V) and current (200 A) as well as a large amount of stored energy (up to 5 kJ) necessary for operation in packet-pulse oscillation modes. The DOMS systems use pulses of long (up to 3 ms) duration (macropulses) formed by packets of short (3–20  $\mu$ s) high-power pulses (micropulses). The short duration of the micropulses allows the formation of electric arcs to be avoided; however, the presence of pauses between them leads to a decrease in the average discharge power and sputtering rate during the macropulse. To implement the arc-free reactive sputtering, the pause duration sufficient for the discharge current decay and compensation for spurious charges accumulated on the target surface and initiating electrical breakdowns are necessary. Using the DU DOMS system allows the formation of long pauses between micropulses to be avoided. Packets of bipolar voltage pulses providing alternative sputtering of the cathodes and automatic compensation for spurious charges are applied to the DMSS cathodes. The minimum pause between bipolar micropulses is determined by the PS and does not exceed 2  $\mu$ s. As a result, the average current and discharge power can be increased without increasing the discharge current amplitudes. In addition, the effective use of the macropulse duration can contribute to an increase in the rate of coating deposition.

To measure the plasma parameters, a triple Langmuir probe placed at a distance of 12 cm from the target surface was used. The triple probe allows the temperature and the electron density to be measured [13]. All potentials of the probe system were close to the floating plasma potential; in this way, high currents cannot damage the probe and disturb relatively weakly the plasma surrounding the probe. Since monotonically varying voltage across individual probes is absent, the plasma parameters can be measured in a very short time. The triple electrostatic probe has low sensitivity to plasma oscillations, since all three electrodes are perturbed simultaneously. During our experiment, the density of the ion current flowing through the substrate was measured with a plane probe equipped with a guard ring. To measure the coating deposition rate, the aluminum films on glass substrates were fabricated. The film thickness was measured with a Linnik-type interference microscope MII-4.

## DISCHARGE PARAMETERS DURING THE MACROPULSE

At the initial stage, the discharge parameters were measured during the macropulse using the Rogowski coil and the triple Langmuir probe (see Fig. 1). The measurements were performed for the following discharge voltage parameters: macropulse duration of 1000  $\mu$ s, macropulse repetition frequency of 10 Hz, micropulse voltage amplitude of 1100 V, micropulse duration of 10  $\mu$ s, and micropulse frequency of 33 kHz. Figure 3 shows the waveforms of the discharge current pulses and the time dependences of the plasma parameters (the saturation ion current density, electron temperature, and plasma density) obtained by processing of signals from the electrodes of the triple probe. On the left plots, the waveforms of the current macropulses and the plasma parameters during the macropulse are shown. The plots

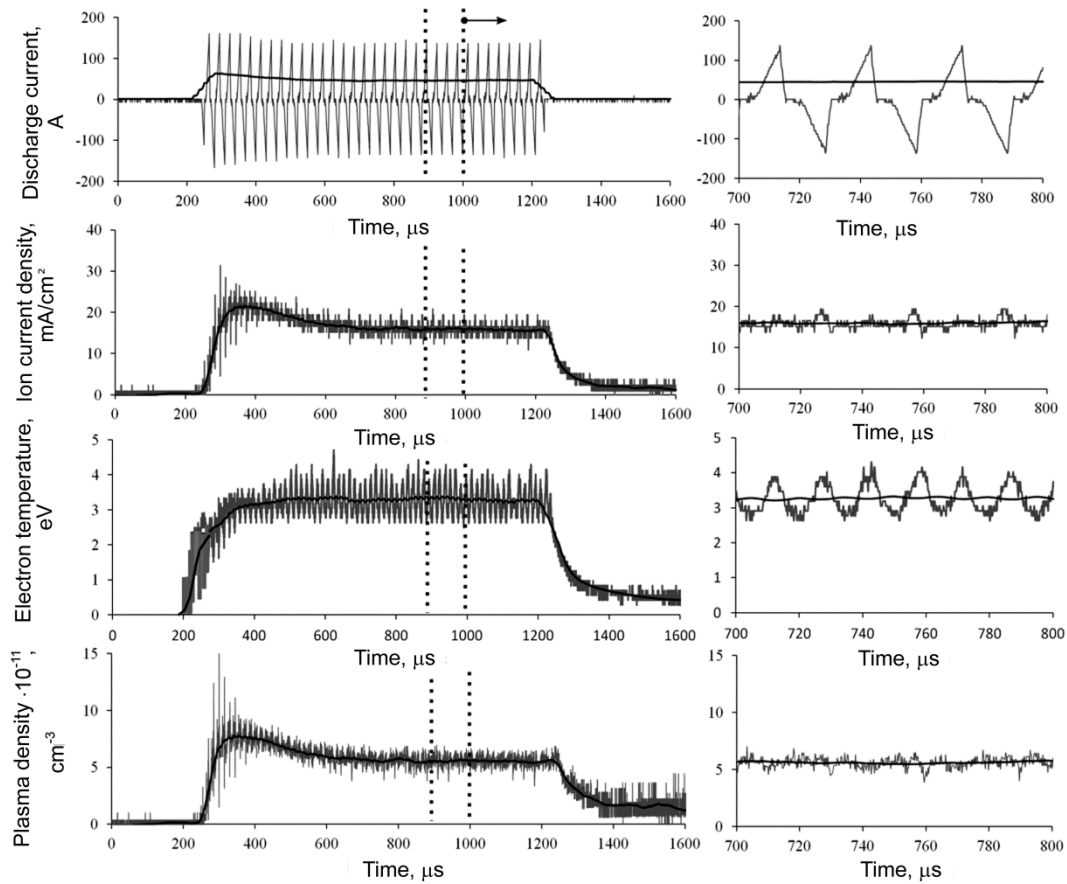


Fig. 3. Time dependences of the discharge current, ion saturation current per probe, electron temperature, and plasma density in the DU DOMS mode.

on the right show the current waveform and the time dependences and the plasma parameters during micropulses. The gray curves show the measured current values of the parameters. The black curves show the average values.

The discharge current pulses had a triangular shape. The discharge did not have time to reach the steady state during the micropulse. At an amplitude of 140–160 A, the average discharge current changed from 50 to 60 A. At the beginning of the macropulse, it reached its maximum. Then due to an increase in the temperature of the working gas its pressure in the near-cathode region decreased [14], thereby leading to a decrease in the discharge current. Therefore, an increase in the plasma density and in the density of ion current flowing to the probe was observed at the beginning of the macropulse. The transient process duration was approximately 400  $\mu\text{s}$ , after which the discharge transformed into the quasi-steady state mode, and the average plasma parameters practically did not change until the macropulse termination.

The continuous ion current flowed to the  $-50$  V bias probe with respect to the grounded chamber. The amplitude of ion current fluctuations did not exceed 15% of its average value. Thus, we can conclude that the ion flux with weakly varying density bombarded the substrate during the macropulse. The electron temperature varied within 2.5–4 eV, and its oscillation frequency coincided with the frequency of formation of the discharge current pulses. The estimated electron density was  $5 \cdot 10^{11} - 7 \cdot 10^{11} \text{ cm}^{-3}$ .

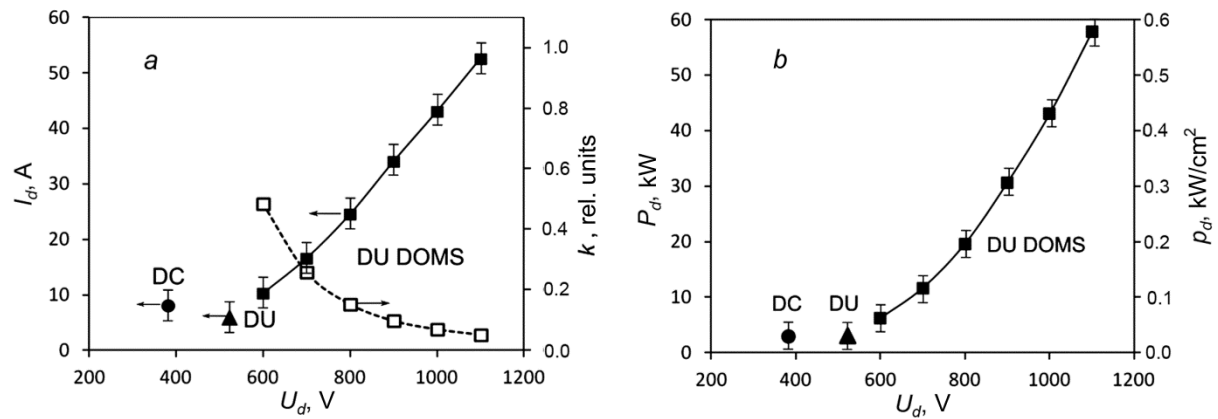


Fig. 4. Dependences of the average discharge current  $I_d$  (a) and of the average discharge power  $P_d$  (b) on the voltage  $U_d$  in the DCMS and DUMS modes and during the macropulse in the DU DOMS mode.

#### CURRENT-VOLTAGE CHARACTERISTIC OF THE PACKET-PULSE MAGNETRON DISCHARGE

In the experiment, the dependences of the current and of the discharge power on the voltage were obtained. The measurements were performed in the DCMS, DUMS, and DU DOMS modes with average discharge power of 3 kW. In the DUMS mode, the pulse frequency was 33 kHz, the duration of the macropulses in the DU DOMS mode was 1000  $\mu$ s, and the pulse repetition frequency varied from 40 to 500 Hz, which corresponded to the change of the macropulse duty factor from 0.5 to 0.04. The micropulse frequency and duration was 33 kHz and 10  $\mu$ s, respectively. Figure 4 shows the dependences of the average current and of the discharge power on the voltage during the macropulse in the DU DOMS mode and of the current and of the discharge voltage in the DCMS and DUMS modes, respectively.

The average discharge current was 8 A in the DCMS mode and 6 A in the DUMS mode. The power density on the target surface for an average discharge power of 3 kW in both modes was approximately 30 W/cm<sup>2</sup>. In the DU DOMS mode, we succeeded in increasing the discharge current up to 10–50 A during the macropulse by decreasing the macropulse duty ratio. Although the average discharge power  $P_d$  remained unchanged, the discharge power during the macropulse  $P_{mp}$  increased by more than ten times from 5 to 56 kW when the pulse duty ratio  $k$  decreased. Wherein, the average power density  $p_d$  on the surface of the DMSS targets increased from approximately 50 to 560 W/cm<sup>2</sup>. Figure 5 shows the dependences of the maximal pulsed discharge current  $I_{max}$  and power  $P_{max}$  on the voltage and of their densities on the DMSS target surface  $J_{max}$  and  $p_{max}$ , respectively. In the DU DOMS mode, the maximal pulsed discharge current reached 160 A, which corresponded to a current density of 3.7 A/cm<sup>2</sup> on the target surface, the maximal pulse discharge power of 170 kW, and the pulse power density of 3.8 kW/cm<sup>2</sup>.

#### DEPENDENCES OF THE PLASMA DENSITY AND ION CURRENT DENSITY ON THE AVERAGE AND PULSE DISCHARGE POWER IN THE DU DOMS MODE

The dependences of the electron density and of the saturation ion current density on the discharge power during the macropulse were measured with the probes. These dependences, as well as the values obtained in the DCMS and DUMS modes for an average discharge power of 3 kW, are shown in Fig. 6. In the DU DOMS mode, the maximal electron density for a discharge power of 56 kW and a power density of 0.6 kW/cm<sup>2</sup> per target was  $7 \cdot 10^{11}$  cm<sup>-3</sup>. The maximal ion saturation current density per probe reached 18 mA/cm<sup>2</sup>. For the same average discharge power, the ion saturation current density in the DU DOMS mode was by a factor of 4.5 higher than in the DUMS mode (4 mA/cm<sup>2</sup>).

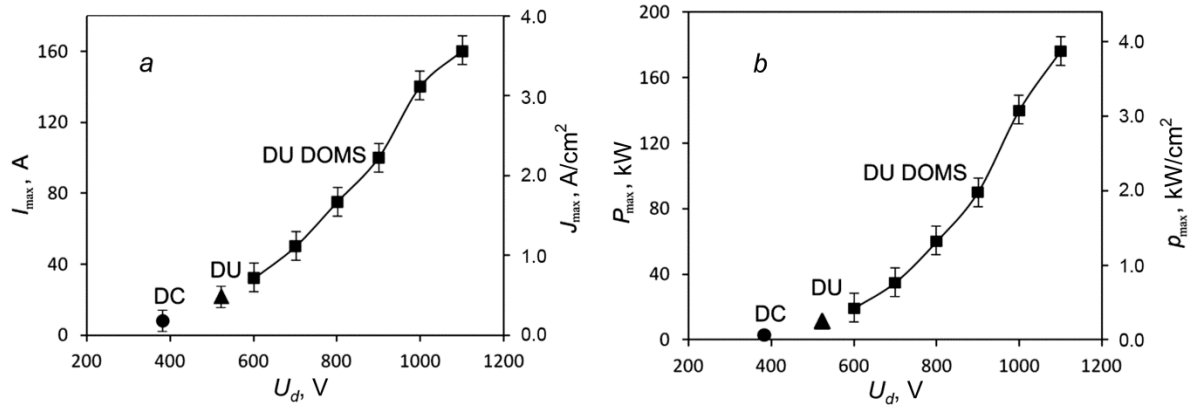


Fig. 5. Dependences of the amplitudes and densities of the discharge current (a) and power (b) on the target surface on the discharge voltage  $U_d$ .

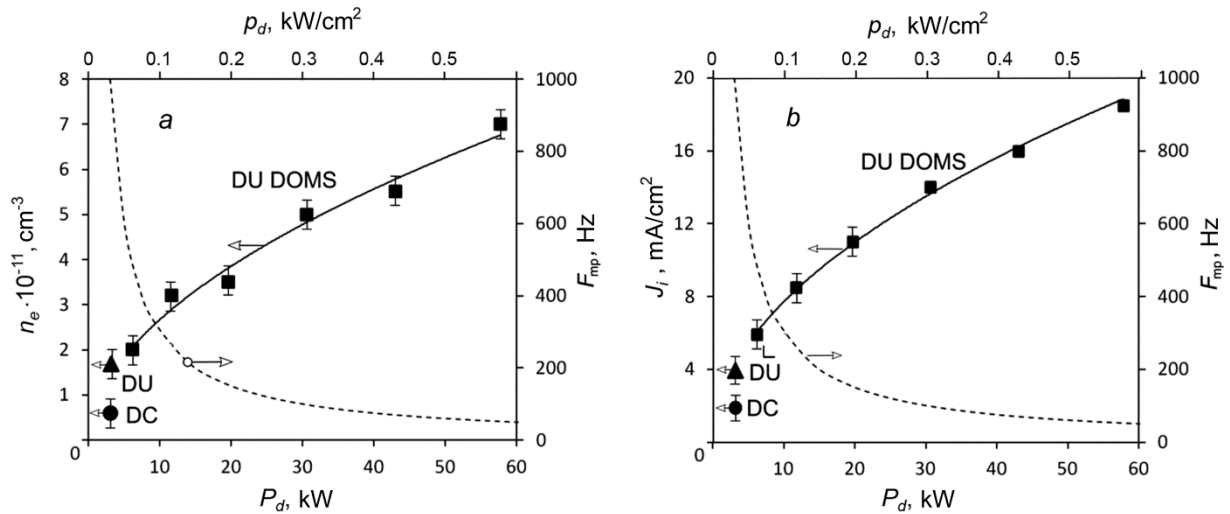


Fig. 6. Dependences of the plasma density  $n_e$  and of the ion saturation current density  $J_i$  per probe on the discharge power.

and by 9 times higher than in the DCMS mode ( $2 \text{ mA/cm}^2$ ). The dotted curve in the plots shows changes in the frequency of the macropulses with the discharge power.

## COATING DEPOSITION RATE IN NON-REACTIVE SPUTTERING MODES

To determine the rate of Al film deposition and the density of the ion flux bombarding the growing coating, a series of experiments was performed in three modes the parameters of which are presented in Table 2. The coatings were deposited on glass substrates at a distance of 85 mm from the target surface. The ion current density was measured with the probe equipped with guard ring installed in place of the substrate.

The results of measurements are shown in Fig. 7. The deposition rate in the DU DOMS mode was by a factor of 3.6 lower than in the DCMS mode and by a factor of 2.8 lower than in the DU mode. A decrease in the deposition rate was characteristic for pulse regimes and was due to the return of a part of the ionized material onto the target [15].

TABLE 2. Magnetron Sputtering Modes

Regime	$U_d$ , V	$I_d$ , A	$I_{mp}$ , A	$I_{max}$ , A	$P_d$ , kW	$P_{Ar}$ , Pa	$f$ , kHz	$f_{mp}$ , $\tau_{mp}$
DCMS	520	8	–	–	3	0.25	–	–
DUMS	380	6	–	22			33	–
DU DOMS	1100	2.5	56 A	160			33	50 Hz, 1000 $\mu$ s

Note.  $U_d$  is the discharge voltage,  $I_d$  is the average discharge current,  $I_{mp}$  is the average current per macropulse,  $I_{max}$  is the maximum pulse current,  $P_d$  is the average discharge power,  $P_{Ar}$  is the working pressure,  $f$  is the micropulse repetition frequency,  $f_{mp}$  is the macropulse repetition frequency, and  $\tau_{mp}$  is the macropulse duration.

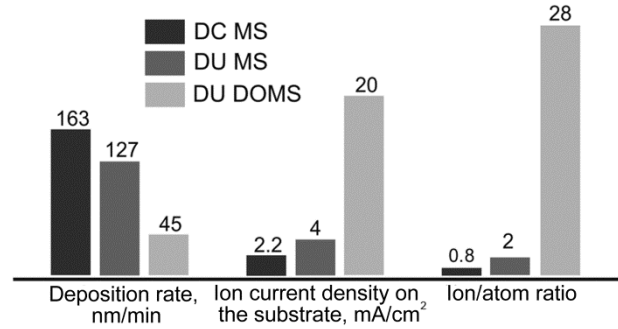


Fig. 7. Diagrams of the Al deposition rate, ion current density on the substrate, and ion/atom ratios depending on the sputtering mode.

The ion current density in the DU DOMS mode reached 20 mA/cm<sup>2</sup>. This value was by 9 times higher than in the DCMS mode, and by 5 times higher than in the DUMS mode.

The change in the degree of ion bombardment of the growing coating can be estimated based on the data obtained. The degree of ion bombardment is characterized by the ratio of the ion flux density  $\Phi_i$  on the substrate to the flux density  $\Phi_{Al}$  of aluminum atoms forming the coating. The flux of aluminum atoms on the substrate surface can be estimated using the coating growth rate  $v$  and the coating density  $\rho$ . For simplicity, we neglect the presence of impurities in the coating and assume that the density of the deposited aluminum coating corresponds to the density of the initial target material:

$$\Phi_{Al} = \frac{v \cdot \rho_{Al}}{m_{Al}},$$

where  $m_{Al} = 4.5 \cdot 10^{-26}$  kg is the aluminum atom mass and  $\rho_{Al} = 2.8 \cdot 10^3$  kg/m<sup>3</sup> is the aluminum density. The ion flux can be estimated from the average ion current density on the substrate (see Fig. 6a):

$$\Phi_i = \frac{J_i}{e},$$

where  $J_i$  is the average ion current density on the substrate and  $e = 1.6 \cdot 10^{-19}$  kg/m<sup>3</sup> is the electron charge. The obtained ratios of the number of bombarding ions participating in the coating formation per each neutral atom are shown in the diagram in Fig. 7.

Calculations showed that a decrease in the coating deposition rate with simultaneous increase in the ion current density on the substrate during the macropulse leads to a multiple increase in the ion/atom ratio. The maximal parameter  $\Phi_i / \Phi_{Al}$  in the DU DOMS mode was by 35 times higher than in the direct current mode and by 14 times higher than in

the dual mid-frequency pulsed magnetron sputtering mode. The parameter  $\Phi_i/\Phi_{Al}$  can be regulated in the range from 0.02 to 0.28 for the given value of the average discharge power by changing the frequency and power of macropulses in the DU DOMS mode. Thus, it is possible to control the degree of ion bombardment of the growing coating to obtain its desired structure.

## CONCLUSIONS

Based on the dual magnetron sputtering system and the pulsed power supply capable of generating high power bipolar packet pulses, the DU DOMS system was implemented. The use of the bipolar pulse voltage the pulses of which were alternately applied to targets of the dual MSS allowed long pauses between micropulses to be avoided and high discharge power with continuous ion current to the high-density substrate to be provided during the entire macropulse. We have succeeded in increasing the discharge power during the macropulse up to 56 kW for an average discharge power of 3 kW and an average power density of 30 W/cm<sup>2</sup>. The pulse power reached 170 kW with pulse discharge current amplitude of 160 A. Such a high discharge power allowed a plasma density of  $7 \cdot 10^{11}$  cm<sup>-3</sup> to be achieved at a distance of 12 cm from the target surface. The probe measurements showed that the ion saturated current density per probe in the DU DOMS mode reached 18 mA/cm<sup>2</sup>. This value was by a factor of 4.5 higher than the ion current in the DUMS mode and by 9 times higher than in the DCMS mode.

Experiments on the deposition of Al films and measurement of the ion current density on the substrate showed that the maximal parameter  $\Phi_i/\Phi_{Al}$  in the DU DOMS mode was by 35 times higher than in the direct current mode and by 14 times higher than in the dual mid-frequency pulsed magnetron sputtering mode. Such an increase in the ion/atom ratio was due to the decrease in the coating deposition rate and an increase in the ion current density on the substrate during the discharge macropulse. The parameter  $\Phi_i/\Phi_{Al}$  showed the degree of the ion bombardment of the growing coating whose structure significantly depends on it. This parameter can be adjusted in the range from 0.02 to 0.28 for the given average discharge power by changing the frequency and voltage.

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