^{04.1} Vacuum (gasless) DC magnetron discharge with a cold planar target-cathode

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A stable mode of a gasless (vacuum) magnetron discharge with a cold planar cathode-target at a residual pressure as low as $4 \cdot 10^{-3}$ Pa and a discharge current of 8-12 A (120-200 mA/cm²) is realized. The results of studies of mass-to-charge ion composition are presented, as well as a comparison of main discharge parameters and a deposition rate in gaseous and vacuum modes.

Keywords: magnetron, vacuum regime, ion composition, deposition rate.

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Magnetron sputtering is a well-known method for deposition of thin metal films with a broad application range. The pressure in the discharge gap is generally kept above $2 \cdot 10^{-1}$ Pa in the process of magnetron sputtering. The results of numerous studies demonstrate that the presence of even an inert gas has a negative effect on the quality and characteristics of coatings deposited by magnetron sputtering [1–3]. In view of this, the issue of reduction of the operating pressure of a magnetron (down to the vacuum (gasless) mode) appears to be relevant to certain technological processes.

A high degree of ionization in a discharge is needed for self-sputtering. The degree of ionization of metal (target material) atoms in a DC magnetron discharge is low (less than 10%) in most cases, and the potential for raising it through an increase in the discharge current is limited by the thermal operating regime of the target. High-power impulse magnetron sputtering (HiPIMS) with a discharge current amplitude of 100 A (or higher) provides an opportunity to produce plasma with the fraction of metal ions being higher than the fraction of working gas ions; the discharge then enters the so-called self-sputtering mode where gas ions do no longer play an essential role in sustaining this discharge [4]. However, such conditions are established only throughout the duration of a discharge pulse. In the mode with a low duty cycle, which is commonly used in high-power impulse magnetron sputtering, metal plasma has enough time to decay between pulses, and a minimum threshold level of the gas pressure in the discharge gap is the only requirement for initiation of the next pulse. In addition, an increase in discharge ignition delay under a low pressure [5] and a corresponding reduction in pulse width and deposition rate necessitate a further increase in gas pressure. Thus, although self-sputtering conditions are established in the HiPIMS mode, the discharge actually remains gaseous in nature.

The feasibility of a planar DC magnetron discharge in the vacuum mode has been demonstrated more than 30 years ago [6,7]. Although this research had application potential, it was discontinued. One of the probable reasons behind this was noted by the authors of these studies themselves: the possibility of additional thermal evaporation from the liquid phase or sublimation of the cathode material under a high discharge power could not be ruled out completely in experiments. Thermal processes contributing to the synthesis of coatings provide a considerable enhancement of their deposition rate. However, in addition to reducing the controllability of coating synthesis, evaporation or sublimation of the cathode material exert a negative (compared to a "purely" magnetron process) effect on the quality of sputtered films, since the energy of thermally evaporated atoms is lower. Notably, the results of our studies demonstrate that the issues regarding stabilization of a magnetron discharge in the vacuum mode have not been resolved completely; this also reduces its application potential.

The purpose of this work is to obtain a vacuum mode of a magnetron discharge with a flat target for studies of the method of high-speed coating deposition. In addition to providing all the advantages over the traditional gaseous mode, a vacuum (gasless) magnetron serves as an alternative to vacuum-arc systems for film fabrication and generation of metal plasma, maintaining a significantly lower level of contamination of plasma and sputtered films by the microdroplet fraction. The primary goals of the present study are to achieve a stable vacuum DC magnetron discharge, examine the mass-to-charge ion composition of plasma, and compare the key parameters of discharge and coating rate in vacuum and gaseous modes.

The diagram of the experimental setup is shown in Fig. 1. A direct water cooling copper target with a diameter of 100 mm was used as a magnetron cathode.



Figure 1. Diagram of the experimental setup.

The target thickness was 4 mm. Since a vacuum (gasless) magnetron discharge is feasible only in the self-sputtering mode, which is established at a certain threshold discharge current density, a stable discharge requires a compromise between increasing the current density by reducing the cathode diameter and the efficiency of removal of heat from a sputtered target, which is enhanced by increasing the mentioned diameter. These factors dictated the choice of the optimum cathode-target diameter, which was set to 100 mm. The target had a developed surface on the back side. The efficiency of heat removal is verified by the fact that the temperature of water discharged from the magnetron did not exceed 22° C at the maximum discharge power of 8 kW, while the temperature of water entering the magnetron frame was $8-10^{\circ}$ C.

An external ring with an outer diameter of 90 mm, which was constructed from 18 NdFeB magnets grade N35 $10\times10\times10$ mm in size, and a center NdFeB magnet grade N35 with a diameter of 20 mm and a height of 10 mm produced a magnetic field. The diameter of the racetrack created in the process of sputtering was 55 mm. The magnetic field strength on the target surface in the racetrack region was on the order of 100 T. A circular electrode with an internal diameter of 100 mm, which had the same potential as the walls of a grounded vacuum chamber and was positioned 8 cm away from the target edge, served as the magnetron anode. This anode positioning was chosen due to the necessity of extending the free path of electrons in the vacuum magnetron discharge mode. The results of experiments revealed that a magnetron discharge is initiated reliably in the gaseous mode at shorter distances, but ceases at pressures below $6 \cdot 10^{-2}$ Pa regardless of the discharge current magnitude.

The magnetron was mounted on the end wall of a vacuum chamber made of non-magnetic stainless steel. A PMI-2 tube mounted at the bottom of the chamber closer to the magnetron was used for pressure measurements. The highest vacuum in the chamber was $2.7 \cdot 10^{-3}$ Pa. The gas flow rate was controlled by an RRG-10 electronic regulator with a mechanical valve, which could close the gas channel completely, inserted in series into the gas line. An APEL-M-20DC-1000SS (OOO "Prikladnaya Elektronika") constant-current source was used as a power supply for the discharge. Films were deposited onto silicon and glass substrates positioned opposite the racetrack at a distance of 10 cm from the target. The deposition time was 15-30 s. The mass-to-charge ion composition in magnetron plasma was examined with a modified quadrupole mass spectrometer based on an RGA-200 residual gas analyzer [8].

In order to get the conditions for vacuum self-sputtering, a magnetron discharge was first initiated in the standard gaseous mode with the working gas admitted into the chamber until a pressure level of $2 \cdot 10^{-1}$ Pa was reached. When the discharge was initiated and the current was raised to 8-12 A, the inflow of gas into the vacuum chamber was cut off by closing the gas line with the mechanical valve. As the pressure in the vacuum chamber dropped down to limit values, no quenching of the discharge was observed at currents starting from 8 A, which corresponded to a mean power density at the target of 75 W/cm^2 . The discharge burning voltage increased as the discharge current stabilized. On average, the gaseous-vacuum mode change resulted in a discharge burning voltage rise with a magnitude of 40-50 V. In the vacuum mode, the magnetron discharge had a weakly increasing current-voltage curve (Fig. 2, a). As the discharge current increased from 8 to 12 A, the discharge burning voltage increased from 594 to 600 V. In the gaseous mode, the magnitude of voltage variation within the same current range was close to 20 V. It should be noted that a change in the magnetron discharge voltage, as well as an increase in pressure in the chamber, was not observed during operation of discharge with duration up to 2 min. Coupled with a near-linear dependence of the deposition rate on the discharge current (Fig. 3), these factors are indicative of a weak influence of thermal evaporation of the cathode-target on discharge processes.

Figure 2, *b* presents the dependences of the gas/metal ion ratio in magnetron plasma in gaseous and vacuum modes on the discharge current. It is worth noting that almost all ions generated in plasma in the examined magnetron discharge scenarios were single-charge. In the gaseous mode, the fraction of ions of the working gas did not exceed 5-10% within the range of currents typical of the vacuum magnetron mode (8-12 A). Conspicuous is the fact that the metal ion fraction in plasma is correlated with the magnetron discharge voltage (Fig. 2, *a*).

Experiments on measuring the rate of deposition of copper films in gaseous and vacuum modes (Fig. 3) revealed that the growth rate of coatings reaches 2μ m/min (a value comparable to the characteristics of vacuum-arc sputtering

systems [9]) when discharge currents and, consequently, the power density in the racetrack region are such a high. It follows from Fig. 3 that the deposition rate in the vacuum mode is 20-30% lower. The coating rate reduction is not offset even by the suppression of transport losses, which are induced by the interaction of sputtered atoms with gas atoms, and the enhancement of sputtering yield due to an increase in the discharge voltage (and, consequently, the ion energy). This effect has not been noted in earlier studies of a vacuum DC magnetron discharge [6,7]. One of its probable causes is the fact that if argon ions are lacking while the current level remains the same, a fraction of sputtered target atoms is forced to return to the cathode after ionization to generate secondary electrons and maintain the discharge. Several research groups have observed a similar effect in the HiPIMS mode when the discharge current increased [10,11]. Although the coating deposition rate decreases slightly, the vacuum magnetron mode has certain advantages: atoms



Figure 2. Current–voltage curve of the magnetron discharge (*a*) and dependence of the ion fraction in magnetron plasma on the discharge current (*b*) for gaseous and vacuum modes. I — Gaseous mode ($p = 2 \cdot 10^{-1}$ Pa) and 2 — vacuum mode ($p = 6.5 \cdot 10^{-3}$ Pa).



Figure 3. Dependence of the rate of deposition of a copper film on the discharge current in gaseous and vacuum magnetron modes. I — Gaseous mode ($p = 2 \cdot 10^{-1}$ Pa) and 2 — vacuum mode ($p = 6.5 \cdot 10^{-3}$ Pa).

of the working gas are not implanted into a forming film, thermalization of sputtered atoms does not occur, and coating may be performed in the collisionless mode ("line-of-sight").

An experimental comparison of the quality of copper films produced with the use of vacuum arc and vacuum magnetron discharges predictably reveals a significantly lower number of defects on the surface in the latter case. However, their presence in the film deposited by magnetron sputtering raises a number of questions. The results of microstructure analysis demonstrated that these defects are identical in composition to the target material, feature no impurities, and have a convex (rather than crater-like) shape, thus excluding the formation of cathode spots on the substrate surface from the list of reasons for defect occurrence. One probable reason on this list is short-term arcing (microarcs) at the magnetron cathode. However, experimental data revealed no dependence of the number of defects on the discharge current. Vacuum purity conditions are another probable reason for the emergence of such defects. This issue requires further investigation.

Nevertheless, the reported research results provide explicit evidence of feasibility of vacuum DC magnetron sputtering with the discharge parameters and characteristics unaffected by the processes of evaporation of the cathode-target material. Coating deposition rates comparable to those typical of vacuum arc systems make a vacuum magnetron discharge a promising alternative technique for high-speed production of quality coatings.

Conflict of interest

The authors declare that they have no conflict of interest.

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