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Magnetron sputtering of tin from a liquid phase in a current stabilization mode

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The possibility of a magnetron sputtering system operation with a liquid-metal cathode is demonstrated in the paper. The experiments with sputtering of a tin cathode from a liquid state in a current stabilization mode were carried out. The coefficient of the cathode sputtering of tin from a liquid state was estimated.

Keywords: magnetron discharge, tin, sputtering.

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It is impossible to imagine modern technology without the use of various methods of application of thin-film coatings. The deposition of hardening coatings on various cutting tools (drills, cutters, millers, etc.) provides an opportunity to reduce wear and extend their service life. Thin films on the surface of different optical parts make it possible to obtain products with the required parameters. The application of coatings with their thickness ranging from several nanometers to tens of micrometers is essential to microelectronics technology.

Magnetron sputtering is currently used widely to apply films of various materials. The target cathode is sputtered in this process with ions of the working gas in a vacuum chamber, and a thin-film coating is thus deposited onto a part [1–5].

The key disadvantage of magnetron sputtering systems (MSSs) is the high energy cost of an atom in the produced coating [6,7]. However, with the cathode being in a liquid phase, one may increase the coating application rate by a factor up to 10 and reduce the energy costs by the same factor while preserving the coating quality. The coating formation rate then becomes comparable to the one typical of vacuum arc evaporation [1].

A low cathode material utilization rate, which is no higher than 40%, is another disadvantage of MSSs with a solid-phase cathode. MSSs with a liquid cathode provide an opportunity to increase the material utilization rate almost to 100% and, consequently, reduce the economic costs significantly and establish waste-free production.

The aim of the present study is to select the processing mode and evaluate the cathode sputtering coefficient and discharge parameters based on the experimental data for a tin cathode sputtered from a liquid phase.

Cathode sputtering was performed using a permanent-magnet magnetron sputtering system modified so as to operate with a liquid-phase cathode. The magnetic system was constructed based on neodymium magnets and installed in a water-cooled steel housing on top of which a crucible with the cathode was positioned. The crucible design provided minimal thermal contact with the water-cooled magnetic system, which resulted in rapid melting of the cathode. The maximum magnitude of the magnetic induction vector component parallel to the cathode on its surface was 0.1 T. The cathode was a grade O1 tin disk with a thickness of 3 mm and a diameter of 55 mm inserted into the steel crucible. The melting point of tin is 232°C.

An MKS Instruments RPG-50 unit with a maximum output power of 5 kW was used as a power supply for the MSS. An IMPAC IP 140 digital infrared pyrometer was used to determine the melt temperature.

The cathode was weighed together with the crucible on a Sartorius CPA225D analytical balance with an accuracy of ± 0.01 mg before and after processing to evaluate the cathode sputtering coefficient.

Figure 1 shows the diagram of the experimental setup. Magnetron sputtering system *3* was positioned in vacuum chamber *1*. The pressure in the chamber was monitored with vacuum gauge *2*. The chamber was grounded; power supply *5* with series-connected ammeter *4* and parallel-connected voltmeter *6* was connected to the MSS and the vacuum chamber. Infrared pyrometer *7* was used to monitor the cathode temperature.

Figure 1, *b* shows the MSS positioned in the chamber. In order to reduce their contamination with tin particles, exposed structural components (cooling tubes and MSS elements) were covered with foil.

Prior to experiments, the vacuum chamber was evacuated to a residual vacuum of $2.5 \cdot 10^{-6}$ Torr within 30 min. Argon was then supplied to the chamber at a flow rate

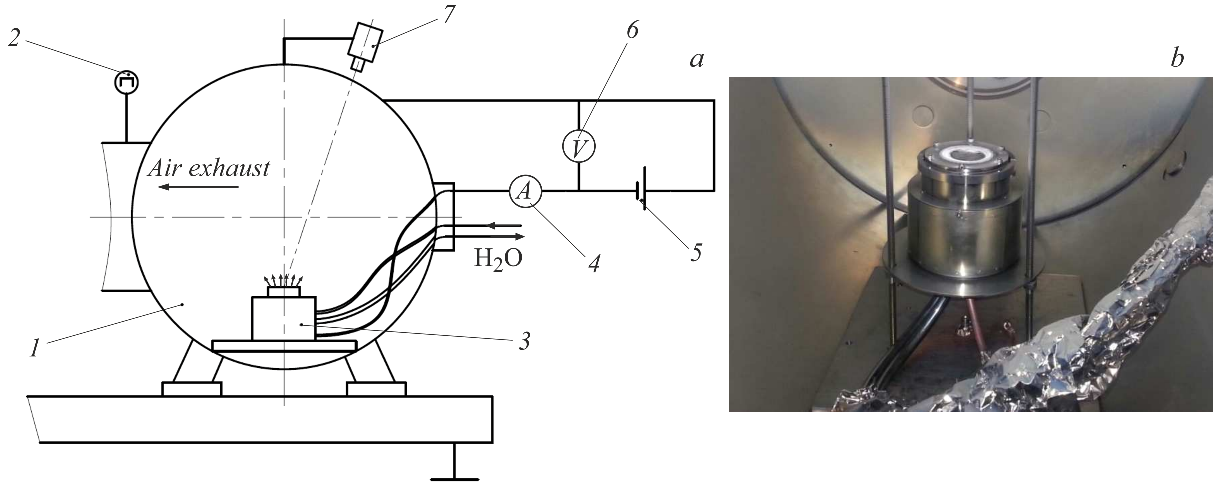

Figure 1. Experimental setup. *a* — Diagram of the setup: *1* — vacuum chamber, *2* — vacuum gauge, *3* — MSS, *4* — ammeter, *5* — power supply, *6* — voltmeter, and *7* — pyrometer. *b* — Photographic image of the MSS in the chamber.

of 100 Nml/min to maintain a pressure at the level of $4.6 \cdot 10^{-4}$ Torr.

The MSS power supply was operated in the current stabilization mode. To minimize the error of determination of the sputtering coefficient, the time of sputtering from a liquid state was made much longer than the time needed for the cathode to melt. The cathode was weighed together with the crucible before and after sputtering to determine the mass of sputtered material. The surface temperature of the melt was measured with the pyrometer after complete melting of the cathode.

The initial heating of the cathode was performed in the mode of power stabilization at a level of 600 W for 5 min. Following complete melting of the cathode, the MSS power supply was operated in the current stabilization mode with a stepwise current growth from 1.1 to 2 A in 0.1 A increments and further to 2.2, 2.5, 3.0, and 3.5 A. A stepwise change in the discharge supply voltage was also observed in the process. Voltage pulsations are attributable to the emergence of sparks on the surface of molten tin in the course of operation of the magnetron sputtering system. Figure 2, *a* presents the time dependence illustrating the dynamics of variation of the magnetron discharge voltage in the process of cathode material sputtering from a liquid phase. The discharge parameters were chosen in such a way that the entire cathode remained in a liquid state and a stable discharge was maintained under a given pressure in the vacuum chamber.

Sputtering from a liquid phase was performed in the current stabilization mode, since it is the most convenient for determining the sputtering coefficient. The entire operation time was 2906 s.

A current–voltage curve of the magnetron discharge for the MSS with a liquid-metal cathode made of tin was plotted (Fig. 2, *b*) based on the experimental data. This current–voltage curve was found to be rising; however, at currents exceeding 2.5 A, the discharge voltage remained virtually unchanged at a level of 710–720 V. In contrast to, e.g., the experiments on sputtering of copper from a liquid phase in [8], self-sputtering of tin could not be achieved at the discharge parameters used.

The pyrometer was used to measure the surface temperature of the molten cathode as a function of discharge power (Fig. 3). This dependence is approximated well by a straight line specified by equation

$$T = 148.5 + 0.122P, \tag{1}$$

where T is the cathode surface temperature [°C] and P is the discharge power [W]. The corresponding R-squared value was $R^2 = 0.9552$, which is indicative of close agreement between the approximating line and the measurement results. Almost all the measured temperature values are above the melting point.

Cathode sputtering coefficient S was determined based on difference Δm between the cathode masses before and after sputtering:

$$S = \frac{e\Delta m}{m_p M_a \sum\limits_i I_i \Delta t_i}, \tag{2}$$

where $e = 1.6 \cdot 10^{-19}$ C is the elementary charge, $m_p = 1.67 \cdot 10^{-27}$ kg is the proton mass, M_a is the atomic mass of the examined material [a.m.u], I_i is the ion current for the i-th time interval [A], and Δt_i is the i-th time interval [s].

Let us present the results of experiments in which the time of operation of the molten cathode in argon was $t = 2600$ s. The initial mass of the cathode with the crucible was 423.4 g, and the mass after sputtering was 414.5 g. Thus, the cathode mass decreased by $\Delta m = 8.9$ g in the course of MSS operation. Using formula (2), we then obtain cathode sputtering coefficient $S = 1.47$.

The rate of cathode erosion in magnetron sputtering from a liquid phase is often found to increase compared

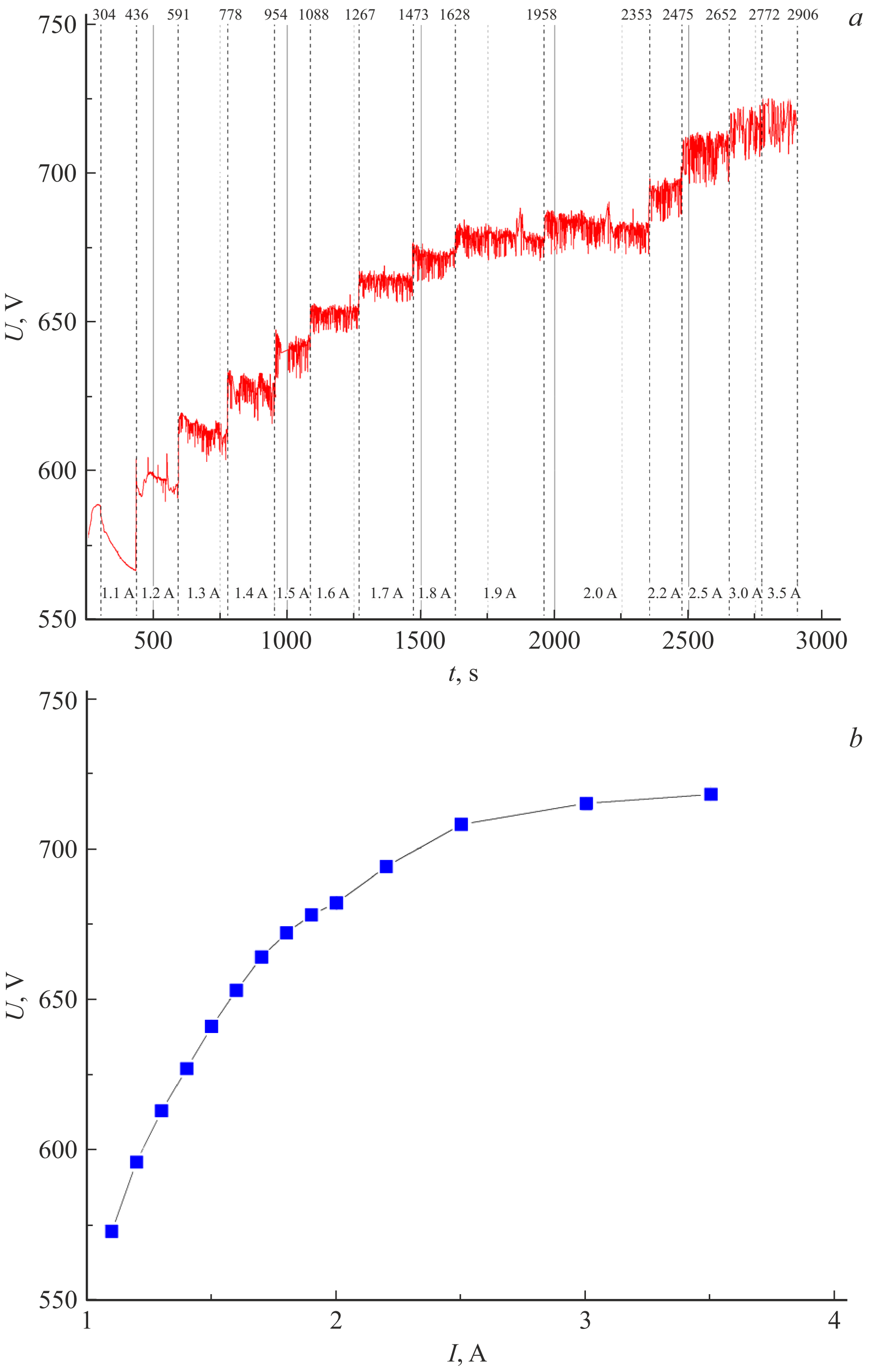

Figure 2. Electrical characteristics of the magnetron discharge. *a* — Dynamics of discharge voltage variation in the current stabilization mode; *b* — current–voltage curve of the discharge.

to the case of sputtering from a solid state. The authors of [9] compared the processes of sputtering of tin from liquid and solid phases and reported an increase in target erosion caused not only by the phase transition, but also by the ejection of tin droplets from the cathode. We have also examined the substrate with tin deposited onto it. No droplets were found on the surface studied under a microscope (with an in-plane resolution up to 110 nm and a vertical resolution up to 10 nm). This may be attributed to the difference in experimental conditions in the present work and in [8].

Thus, the current–voltage curve for a magnetron discharge on molten tin was plotted. It follows from the analysis of the obtained dependence that the voltage varies only slightly within the range of 710–720 V at discharge currents exceeding 2 A.

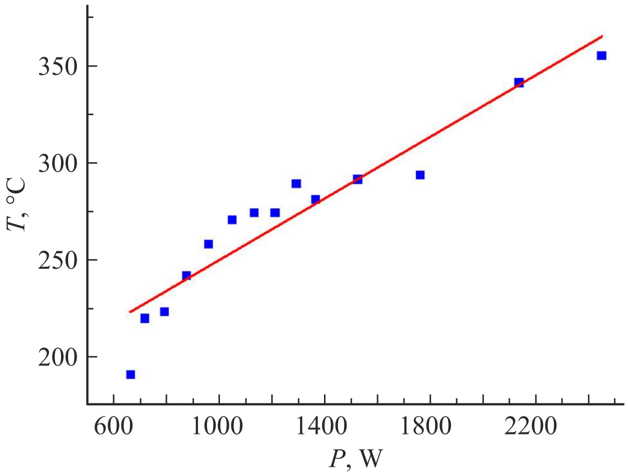

Figure 3. Dependence of the cathode temperature on the discharge power.

The dependence of surface temperature of molten tin on the discharge power was plotted. This dependence is approximated well by a straight line. Coefficient of sputtering $S = 1.47$ of tin from a liquid phase was determined based on the experimental data.

Conflict of interest

The authors declare that they have no conflict of interest.

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