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Deposition rate and substrate temperature during deposition of silicon-carbon coatings by electron beam evaporation of silicon carbide

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Silicon-carbon coatings are prepared by electron beam evaporation of silicon carbide. The heating of the substrates occurred due to the thermal radiation of the target. The deposition rate and the substrate temperature are inversely proportional to the square of the distance to the target. It is shown that the deposition rate is uniquely related to the substrate temperature.

Keywords: Electron beam evaporation of silicon carbide, silicon-carbon coatings, deposition rate, thermal radiation, substrate temperature.

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Introduction

The properties of silicon-carbon films deposited by electron-beam evaporation allow for their use as protective and hardening coatings [1]. One of the main advantages of electron-beam evaporation is the higher deposition rate compared to the plasma-enhanced chemical method [2] and magnetron sputtering [3]. The problem of substrate heating by all types of radiation during magnetron sputtering was addressed in [4, 5]. At the same time, substrate heating is considered [6] insignificant during electron-beam evaporation, which allows depositing coatings on heat-sensitive substrates. However, as for silicon carbide evaporation, deposition rates of the order of 1 $\mu\text{m}/\text{min}$ and higher are achieved by heating the target to temperatures over 2500 K. This is accompanied by intense thermal radiation, which leads to noticeable heating of substrates. As for substrates

made of low-melting materials, for example, aluminum, such heating can result in fatal consequences. The aim of this paper is to analyze the relationship between the coating deposition rate and the substrate heating temperature.

Experimental technique and methodology

To establish the relationship between the substrate temperature and the coating deposition rate, experiments were conducted using a setup equipped with a fore-vacuum electron source with a plasma cathode [7]. The working chamber accommodated the tooling, which included a graphite crucible with a target placed inside it. The target consisted of a pellet 12 mm in diameter and 3 mm thick, sintered from silicon carbide powder with a grit size of F 60 (Fig. 1). Five substrate holders, placed on steel supports at various distances from the target, shared the same platform with the crucible.

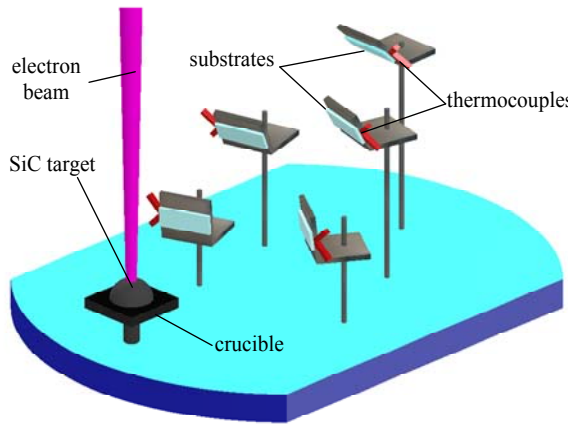


Fig. 1. Schematic of silicon-carbon coating deposition

Borosilicate glass plates sized $10 \times 20 \times 2$ mm were used as substrates. The solid angle within which the substrates were located did not exceed 0.2 steradians. A chromel-alumel thermocouple was attached to each substrate.

Deposition was carried out in a helium atmosphere continuously for one hour. The thickness of the deposited films was measured using an MII-4 microinterferometer.

Measurement results

The experimental dependencies shown in Fig. 2 demonstrate that, within the margin of error, the deposition rate ν and the substrate temperature T_s are inversely proportional of the square of distance r from the target to the substrate at a constant electron beam power. Moreover, as the electron beam power increases, the slope of the ν versus $(1/r^2)$ dependences increases, while the slope of the $T_s(1/r^2)$ straight lines changes less significantly. The dependence of the deposition rate on the substrate temperature (Fig. 3) is of particular interest. Despite the fact that the measurements were performed for substrates located at different distances from the target, the experimental points fit reasonably well onto a single curve. For a low-melting substrate material, such as aluminum, this dependence means that in the absence of forced substrate cooling, the deposition rate has a limit set by the substrate temperature.

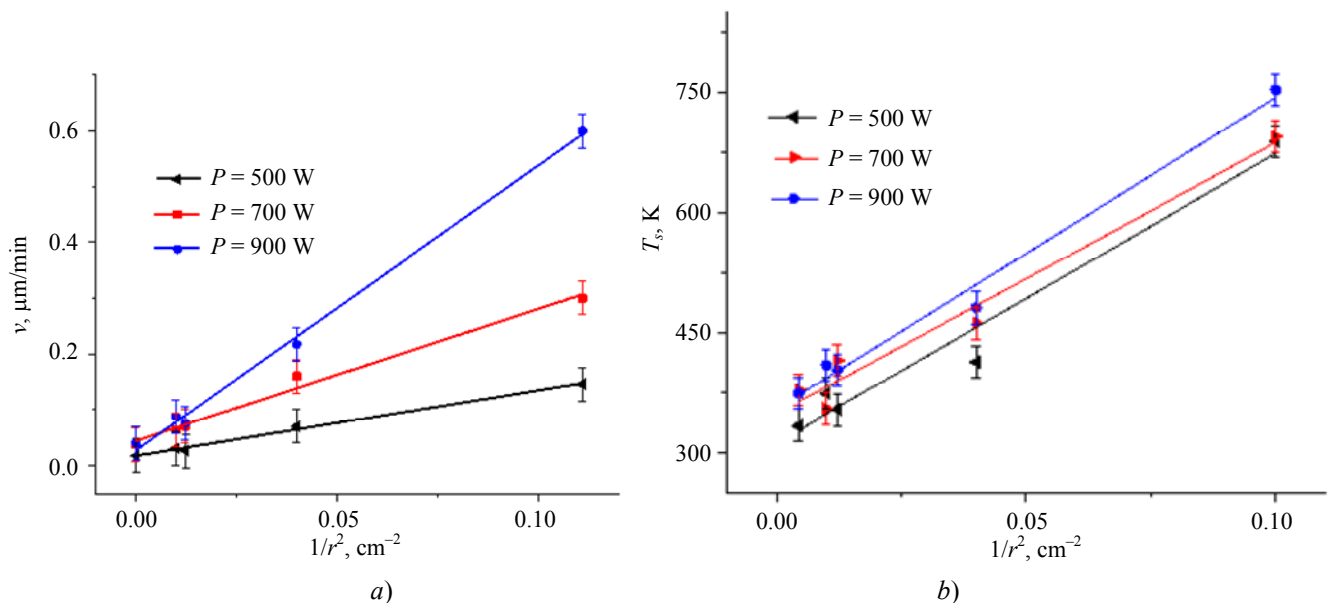


Fig. 2. Deposition rate ν (a) and substrate temperature T_s (b) as a function of the inverse square of distance $1/r^2$ for different electron beam powers

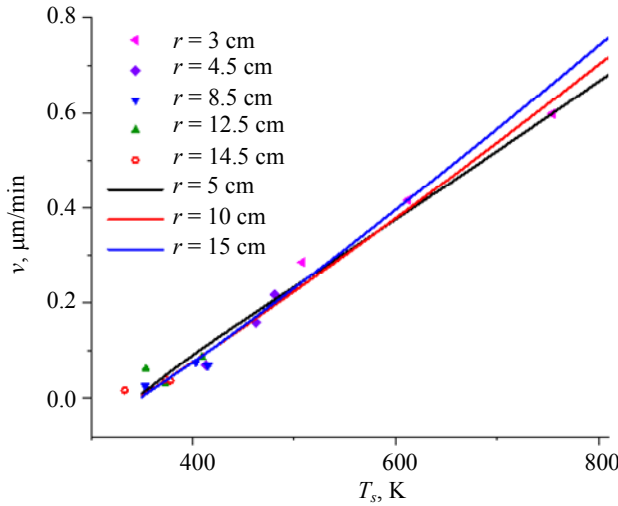


Fig. 3. Experimental (points) and calculated (lines) dependences of deposition rate v on substrate temperature T_s

Analysis of thermal load and deposition rate

To explain the obtained dependences, we adopt the following assumptions. Evaporated particles travel without collisions with gas molecules and are deposited on the surfaces they reach. The target is a black body. Silicon carbide evaporation follows the Clausius-Clapeyron law with the Antoine correction [8]. Let's define evaporation rate V_{ev} as the product of the saturated vapor density, average molecular velocity and evaporation area S_t :

$$\begin{aligned} V_{ev} &= S_t \sqrt{\frac{kT_t}{m}} \frac{mp_0}{kT_t} \cdot e^{-\frac{\Delta H}{R(T_t+C)}} = \\ &= S_t \sqrt{\frac{\mu}{RT_t}} p_0 \cdot \exp\left[-\frac{\Delta H}{R(T_t+C)}\right] \end{aligned} \quad (1)$$

where T_t is the target temperature; m is the mass of SiC molecule; μ and ΔH are the molar mass and heat of evaporation of silicon carbide; R is the gas constant; C is Antoine correction; p_0 and C are constants determined by comparison with experiment. Considering the assumptions listed above, we obtain $V_{dep} = V_{ev}/2\pi r^2$ for deposition rate V_{dep} . The target temperature is related to power P_t delivered to the target by electron beam $T_t = (P_t/S_t\sigma)^{1/4}$, where σ is the Stefan-Boltzmann constant. To establish the relationship between substrate temperature T_s , substrate area S_s , and target temperature T_t , it is necessary to use the setting mechanism of substrate thermal balance. If the energy loss by the substrate

occurs via thermal radiation, then from the energy balance equation:

$$P_t \frac{S_s}{4\pi r^2} = 2S_s\sigma T_s^4, \quad (2)$$

$$\text{it follows that } T_s = \left(\frac{P_t}{8\pi\sigma r^2}\right)^{1/4} \quad (3)$$

That is, substrate temperature T_s depends on distance as $1/r^{1/2}$. This does not correspond to the experimental dependence shown in Fig. 2. Another heat removal path is thermal conductivity. Under the condition that P_t is on the order of one kilowatt, heat flux P_s to the substrate estimated from expression $P_s = \frac{P_t}{4\pi r^2} S_s$ amounts to a few watts. At the pressure of several pascals, the thermal conductivity of gas cannot provide such heat removal. Consequently, the main heat flow path is the thermal conductivity through the substrate holder elements. Since precise calculations are hardly appropriate, the heat flux from the substrate can be accounted for by expression $\alpha(T_s - T_b)$ [4], where T_b is the temperature of the platform on which the substrate holder is mounted, and coefficient α is to be determined from comparison with experiment. Estimates can be made to confirm the possibility of heat removal from the substrate via thermal conductivity. Heat flow Q through a steel rod of diameter d and length l , on which the substrate holder is placed, can be written as:

$$Q = \frac{\lambda \pi d^2}{4l} (T_s - T_b), \quad (4)$$

where $\lambda = 45$ W/m·K is thermal conductivity of steel. For $d = 3$ mm, $l = 5$ cm, $T_s - T_b = 400$ K we have $Q = 2.5$ W, which is consistent with the radiant energy flux to the substrate. Considering the assumptions made, the energy balance equation on the substrate now takes the form:

$$S_t \sigma T_t^4 \frac{S_s}{2\pi r^2} = \alpha (T_s - T_b), \quad (5)$$

That is, the substrate temperature is inversely proportional to the square of the distance. Expressions (1) and (5) reflect the relationship between the coating deposition rate and substrate temperature with the target temperature, and hence with the electron beam power. By expressing T_t from (5) and substituting it into (1), and also considering the relationship between the rates, we obtain an expression convenient for analyzing the dependence of deposition rate V_{dep} on substrate temperature T_s :

$$V_{dep} = \frac{A}{r^{9/4} (T_s - T_b)^{1/8}} \times \exp \left\{ -\Delta H / R \left[B \sqrt{r} (T_s - T_b)^{1/4} + C \right] \right\}, \quad (6)$$

$$\text{where } A = \frac{S_t}{2\pi\sqrt{B}} p_0 \sqrt{\frac{\mu}{R}}, \quad B = \left(\frac{2\pi\alpha}{\sigma S_t S_s} \right)^{1/4}.$$

At first glance, the dependence of the deposition rate on distance that follows from expression (6) appears as $V_{dep} \sim r^{-9/4}$, which

differs from $V_{dep} \sim r^{-2}$ found experimentally. However, one should consider the presence of r in the exponent of (6), according to which V_{dep} weakly increases with increasing r . The calculated curves plotted in relative units (Fig. 3) match the experimental points. Moreover, the curves obtained for different distances slightly differ from each other, which also corresponds to the experimental results. The slight change in the slope of the lines in Fig. 2b cannot be satisfactorily explained within the framework of the proposed model. However it can be noted that in the same range of distance r variation, deposition rate V_{dep} changes by an order of magnitude, while substrate temperature T_s changes by only a factor of two.

Conclusion

The performed measurements and analysis have shown that substrate heating due to thermal radiation from the target can be significant during deposition of silicon-carbon coatings by electron-beam evaporation of silicon carbide. In the case of a low-melting substrate material and absence of forced cooling, this heating imposes limitations on the electron beam power and, consequently, on the coating deposition rate. Moreover, the deposition rate is unambiguously determined by the substrate temperature. This limitation should be expected during deposition of coatings from other materials with a high evaporation temperature.

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