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Electron beam evaporation of alumina ceramics at forevacuum pressures

The paper presents research results on electron beam evaporation and vapor deposition of alumina ceramics using a plasma electron source designed specially for operation at forevacuum pressures. The deposited coating thickness is analyzed depending on the distance to the evaporated target and on the angle between the beam axis and the direction to the substrate center. It is shown that in the forevacuum pressure range, the spatial distribution of the coating thickness is more uniform compared to deposition in high vacuum. This fact can be associated with dispersion of evaporated material particles at gas molecules.

Keywords: electron beam evaporation, plasma electron source, forevacuum pressure range, dielectric surface charging.
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One of the major features of electron beam evaporation of materials is the possibility to attain high power densities in which the electron beam far excels other heat sources [1, 2], being second, in some cases, only to laser radiation. Concentration of comparatively high power over a small area provides local heating of materials, which is particularly important for treatment of refractory metals and alloys with minimal changes in their initial structure in the exposure zone. Electron beam treatment of non-conducting (dielectric) materials, compared to metals, has specific features associated primarily with the necessity to neutralize the charge brought by an electron beam to an irradiated non-conducting surface [3, 4]. One of the ways of solving the problem is to use forevacuum plasma electron sources capable of producing electron beams in the range of pressures higher than those typical of standard electron sources. In a forevacuum electron source operating at 5–20 Pa, the negative surface charge is compensated both by ions from the beam plasma and by ions from the plasma of a non-self-sustained discharge arising between an irradiated target and grounded walls of the vacuum chamber [5]. The efficiency of energy transport from an electron beam to an irradiated dielectric target in the forevacuum pressure range has been demonstrated on the example of welding and melting of ceramic materials [6, 7] and their surface hardening [8]. Another application of electron beams at forevacuum pressures can be evaporation of non-conducting materials, in particular ceramics, with subsequent vacuum deposition of coatings. When arrived at an evaporated target, the accelerated electrons of a beam transfer their kinetic energy to the target, heat its thin surface layer of depth no greater than several micrometers, and provides evaporation right from the material surface, making these heating method more efficient than others. The possibility of direct electron beam treatment of dielectric materials is the key feature of forevacuum plasma electron sources. Moreover, it is possible to realize evaporation of dielectrics in the sources without a crucible, which can greatly increase the evaporation efficiency. The aim of our study was to realize and investigate electron beam evaporation of dielectric materials, in particular ceramics, in the forevacuum pressure range.

Experiment

In the experiment, we studied how the thickness of deposited film depends on the spatial location of a substrate on which it is deposited. The substrates were located at different distances from the evaporated target and at the same distance but at different angles to the beam axis. A schematic of the experimental arrangement is shown in Fig. 1.

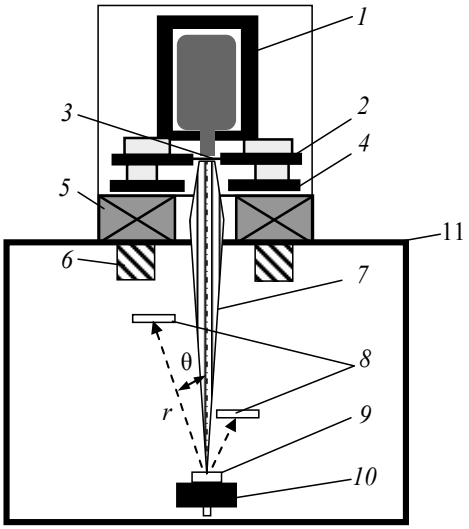

Fig. 1. Experimental arrangement: *1* – cylindrical hollow cathode; *2* – anode; *3* – emission electrode (perforated plate); *4* – accelerating electrode-extractor; *5* – focusing solenoid; *6* – deflecting magnetic system; *7* – electron beam; *8* – substrates; *9* – evaporated specimen; *10* – graphite holder; *11* – vacuum chamber

The plasma electron source was based on a hollow-cathode discharge and produced a narrow focused electron beam in forevacuum. The operating pressure in the vacuum chamber was 10 Pa and was controlled by gas supply directly to the chamber. The source was operated in isobaric mode, i.e., with no gas supply to the cathode cavity. This mode is typical for forevacuum plasma electron sources.

The evaporated material was alumina ceramics (VK94-1) shaped as a disk of diameter 14 mm and height 5 mm. The evaporated specimen was placed in the cavity of a graphite crucible located in the vacuum chamber in the plane of beam incidence (on the beam axis).

The beam current sufficient for evaporation of ceramics was ensured by emission through 120 holes of diameter 0.7 mm in the emission electrode (anode) of the source. At an accelerating voltage of 10 kV and beam current of 50 mA, the power density at the target reached 800 W/cm^2. The substrates were made of glass to dimensions of 15×20 mm and thickness of 2 mm and were arranged at distances multiple of 2.5–3 cm from the evaporated target and at a fixed angle between the beam axis and the direction to the substrate center (Fig. 1). The distance to the first and the last substrate was 4.5 and 19.5 cm, respectively. Before evaporation, the target was uniformly heated by a defocused electron beam for 10 min to preclude fracture of the specimen under thermomechanical stresses during electron beam irradiation. Within 20 min after, the electron beam was focused at the specimen, and at a power density of 600 W/cm^2, intense evaporation of the ceramics began. The evaporation time at constant beam power density was 20 min.

One of the main parameters characterizing the evaporator is the spatial distribution of evaporated material vapors. The spatial distribution was determined by measuring the deposited film thickness on the substrates uniformly arranged on the inner surface of a semi-sphere with the evaporated material at its center (Fig. 2) for which we used a MII-4M interferometer.

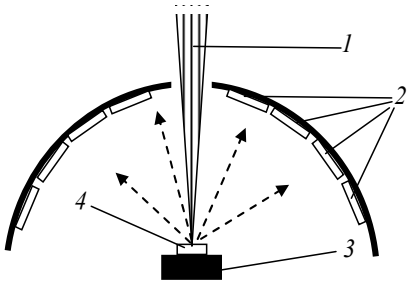

Fig. 2. Arrangement for measuring the angular distribution of evaporated material: *1* – electron beam; *2* – substrates; *3* – graphite crucible; *4* – ceramic specimen

Thus, we constructed an angular distribution of the film thickness knowing its measured values and the angle between the beam axis and the direction to the substrate center. The elemental composition of the film was analyzed with a Quanta 200 3D-scanning electron microscope (EDAX, Netherlands).

Results and analysis

Evaporation in high vacuum has been rather thoroughly studied; electron beam evaporation from different types of sources (point, wire, two-dimensional) is considered elsewhere [9]. For evaporation from a two-dimensional source, as is in the experiment, the distribution of the deposited layer thickness obeys Knudsen's cosine law [10]:

$$h = \frac{m}{\pi\rho} \cdot \frac{\cos^2\theta}{r^2}, \qquad (1)$$

where h is the film thickness on a substrate, m; m is the mass of material evaporated per unit time, kg/s; ρ is the density of evaporated material, kg·m^{-3}; r is the distance from the source to the substrate, m; θ is the angle between the normal and the direction to the substrate center.

According to this law, the matter deposited per unit area per unit time is inversely proportional to the squared distance from the source to the substrate.

Our experiments demonstrate that for electron beam evaporation in the forevacuum pressure range, the deposited film thickness decreases monotonically with increasing the distance h from the evaporated material to the substrate (Fig. 3); the experimental points rather accurately fit the straight line calculated by the cosine law (1). The evaporation rate in the experiments was 20–80 μg/cm^{2}s, and the deposition rate was 2.6–10.4 μg/cm^{2}s. Increasing the gas pressure decreased both the ceramic evaporation rate and the film deposition rate.

Another distinguishing feature of electron beam evaporation at forevacuum pressures is a more uniform angular distribution of evaporated material.

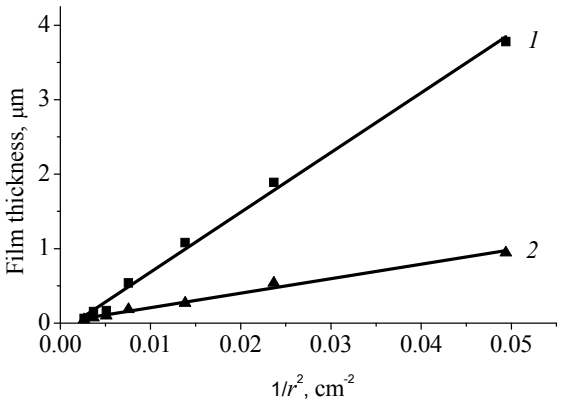

Fig. 3. Film thickness h vs the squared distance to the source at 10 Pa for helium (*1*) and oxygen (*2*).

Figure 4 presents measurement results for the film thickness on the substrates uniformly arranged on the inner surface of a semi-sphere with the evaporated material at its center. Most of the evaporated material reaches the substrate and is rather uniformly deposited up to angles of ±45° with the beam axis (Fig. 4).

For comparison, the same figure shows an angular distribution calculated by the cosine law (curve *3*). Varying the electron beam parameters scarcely affects the distribution.

The elemental composition of the film (Table) obtained by evaporation of the alumina ceramics in helium and oxygen shows that in the inert gas, the film is depleted of oxygen. The ratio of oxygen to aluminum is 0.78, whereas this ratio in the evaporated material is 1.5.

The situation can be changed by adding oxygen in the vacuum chamber. In this case, the elemental composition of the film remains unchanged but the atomic

ratio between oxygen and aluminum becomes close to the ratio in the evaporated material.

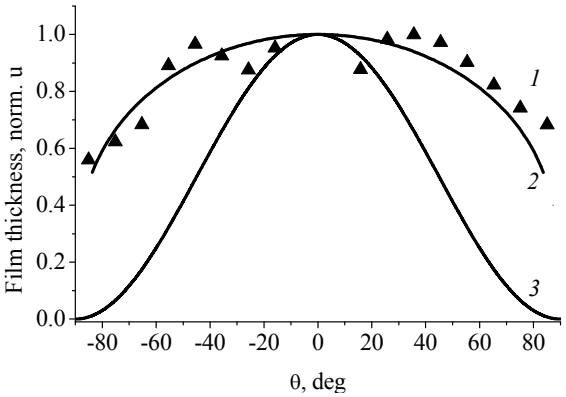

Fig. 4. Angular distribution of the film thickness *h*: *1* – experimental data for a power density 600 W/cm^2 and pressure of 8 Pa; *2* – approximation of the experimental data; *3* – calculations by the cosine law

Elemental composition of the coating

Element	Evaporation in oxygen	Evaporation in helium
	Relative content, %	Relative content, %
C	11.83	22.43
O	47.56	33.21
Al	31.25	42.55
Si	9.36	1.81

Conclusion

Electron beam irradiation of ceramic materials at forevacuum pressures allows their evaporation with subsequent vapor deposition of coatings without negative charge accumulation at the irradiated surface. The thickness of thus obtained coatings is more uniform compared to deposition in high vacuum. This fact can be explained by dispersion of evaporated material particles at gas molecules under high pressure.

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Особенности электронно-лучевого испарения керамики на основе оксида алюминия в форвакуумной области давлений

Представлено исследование процесса электронно-лучевого испарения и осаждения из паровой фазы керамических материалов в форвакуумной области давления. Для испарения алюмооксидной керамики на основе оксида алюминия использовался плазменный электронный источник, специально созданный для работы в форвакуумной области давлений. Определена зависимость толщины напыляемого покрытия от расстояния до испаряемой мишени и угла между осью электронного пучка и направлением на центр подложки. Показано, что в форвакуумной области давлений пространственное распределение толщины покрытий более равномерное по сравнению с напылением в высоком вакууме. Это обстоятельство может быть связано с рассеянием частиц испаренного вещества на молекулах газа при высоком давлении.

Ключевые слова: электронно-лучевое испарение, плазменный электронный источник, форвакуумная область давлений, зарядка поверхности диэлектрика.