Electron-beam Deposition of Ceramic Coatings

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Abstract

This paper presents a novel technique for producing alumina ceramic coatings based on the evaporation of an aluminum oxide target by an electron beam in the middle vacuum range (5-30 Pa). The evaporation of ceramics by the electron beam enables to attain a coating deposition rate of up to 0.3 μm/min, and thus consider this as a practical alternative to existing methods. A special attention is devoted to the study of the surfaces of obtained coatings. The X-ray fluorescence analysis of the deposited coating showed the presence in the coating of all the elements that contained in the ceramic target being evaporated. The coating has a substantially homogeneous surface without any prominent pores. Copyright © VBRI Press.

Keywords: Plasma, electron beam, fore-vacuum.

Introduction

Ceramics as a construction material has gained a wide application in modern science and technology [**1-2**], with electric engineering and electronics being its major industrial consumers [**3**]. Contemporary ceramic materials are demanded to possess high mechanical and electrical strength, vacuum tightness and to be thermally, chemically and radiation resistant. Ceramic materials based on aluminum oxide $A1_2O_3$ most closely meet these requirements and have become indispensable construction materials for the instrumentation industry of today. Currently, the most widely used methods of ceramic coating deposition are gas-thermal evaporation, vacuum-arc deposition, and vacuum evaporation [**4**]. We propose an alternative method for producing coatings based on the evaporation by an electron-beam and subsequent deposition occurring in fore-vacuum. This method is unique in that it combines the beam and plasma techniques, because in the fore-vacuum range of pressure (1-100 Pa), the electron beam generates plasma, which greatly alters the interaction picture of both between the electron beam and the ceramic target and between the flowing evaporated material and the substrate. The propagation of the electron beam in the given range of pressure is accompanied by the generation of plasma, which eliminates the problem of charge accumulation on the dielectric target and, additionally, increases the gas reactivity and preserves the surface stoichiometry, which is especially important for the deposition of ceramic layers.

This method has its advantage in simplicity of implementation, sufficiently fast deposition rate, and the possibility to vary the coating parameters in a wide range [**5**].

Experimental

Setup for electron beam evaporation

The experiments in ceramic evaporation were conducted using a fore-vacuum plasma electron source based on the hollow cathode glow discharge operating in continuous mode. The source 1 was placed on the upper flange of the vacuum chamber 11 (**Fig. 1**) and generated the electron beam 3 of a current of 200 mA and energy of 1-20 keV.

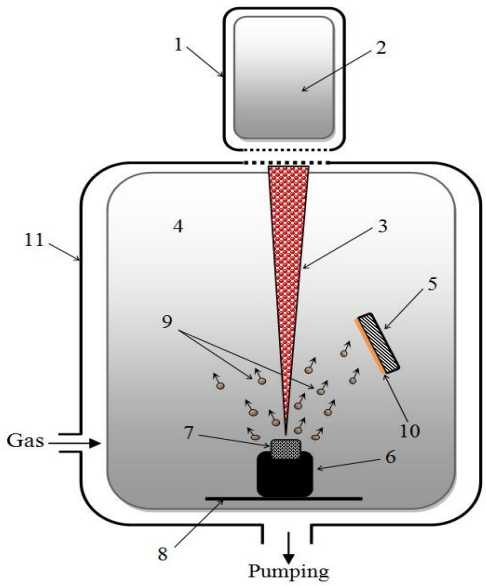

Fig. 1. Experimental setup: 1 – plasma electron source, 2 – discharge plasma, 3 – electron beam, 4 – gaseous beam-produced plasma, 5 – substrate, 6 – crucible, 7 – ceramic target, 8 – collector, 9 – plasma and metal vapor from ceramic target, 10 – deposited coating, 11 – vacuum chamber.

The electrons were extracted from the glow discharge plasma 2, having a burning voltage of 200–500 V and a current of 100–500 mA. The beam was focused to a diameter of 3 mm by the magnetic field of the focusing system. The electron collector 8 was a 70 mm steel disk. The tantalum crucible 6 with an aluminum oxide ceramic sample 7 was placed on the collector. The electron beam, when traveling to the ceramic target through the vacuum chamber filled with the working gas at a pressure of 1–15 Pa, generated a dense beam plasma 4. The effect of the electron beam on the target 7 resulted in its heating, evaporation and partial ionization. The surface charge of the target was neutralized owing to the ions being deposited on its surface from the beam plasma 4. The material evaporated from the target surface deposited on the experimental sample 5 (substrate) whose surface was at a 30 degree angle with the beam's propagation axis.

A picture of the beam, target, and collector is shown in **Fig. 2**.

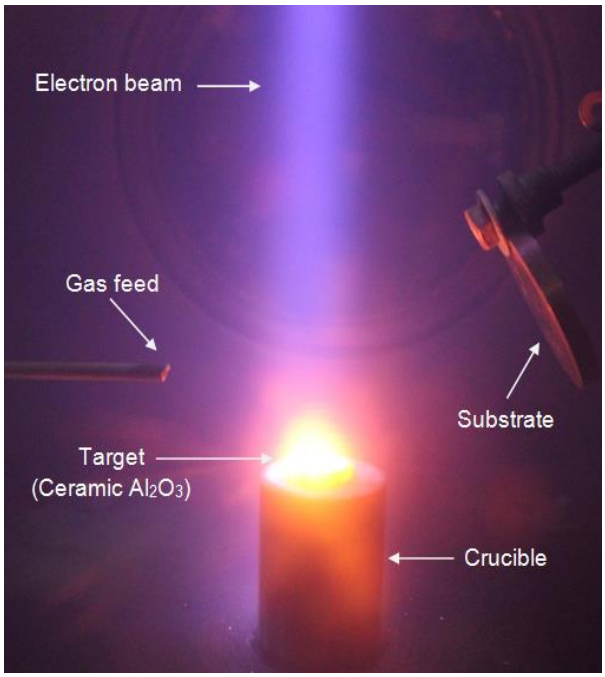

Fig. 2. Picture of the coating deposition process.

The deposition of ceramic atoms and ions on the surface of the target resulted in the formation of coating 10. The distance from the beam's axis to the substrate was 5 cm. The duration of the whole process was 5 minutes.

Plasma and coatings diagnostics

Mass-to-charge composition of ions of beam plasma produced as result of electron-beam of evaporation of ceramic target, was monitored using modified RGA-100 gas analyzer [**6**]. The picture of the coating was shot using the raster electron microscope Hitachi S3400N coupled to the EDS Bruker X'Flash 5010.

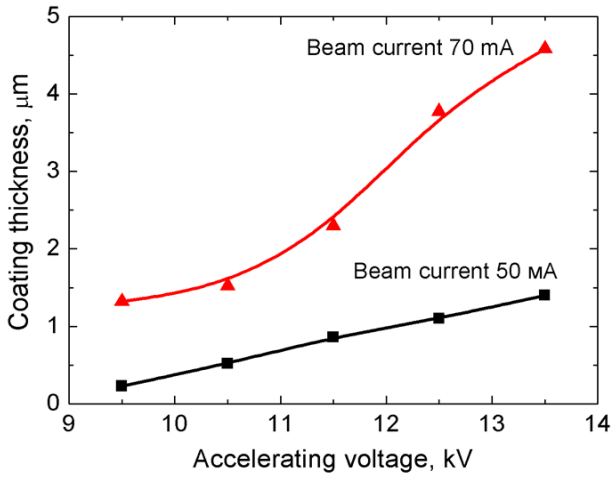

Fig. 3. Dependence of the ceramic coating thickness on the electron beam parameters.

Results and discussion

We measured the evaporation and coating rates versus the beam current and energy. The rate of evaporation from the ceramic target was about 70 mg/min (about 4 g/hour). The rate of the substrate coating was about 0.3 μm/min. The dependence of the coating thickness on the beam parameters is shown in **Fig. 3**. Based on these data, we determined the deposition times for ceramic coating.

Fig. 4 shows the mass-charge spectra of plasma ions for aluminum oxide ceramics during heating (red) and evaporation (black). As seen, at the initial moment, when the target is being heated up by the electron beam, the spectrum is characterized mostly by the presence of the ions of the residual atmosphere.

However, under intensive evaporation of ceramics, there began to appear in the spectrum the ions K^+, Al^+, $Al(OH)^{2+}$, $Al(OH)^+$, Na^+. All these elements are constituent parts of aluminum oxide ceramics. This testifies to the fact of effective ceramic evaporation and formation of secondary ceramic plasma, which subsequently deposits on the substrate and thus forms coating.

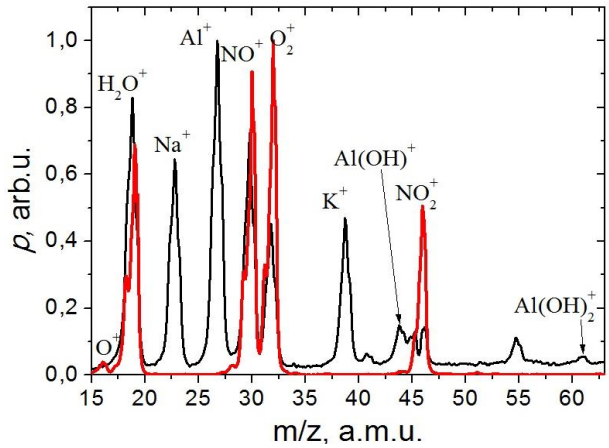

Fig. 4. Typical spectra recorded by the modified quadrupole mass analyzer RGA-100 [**6**].

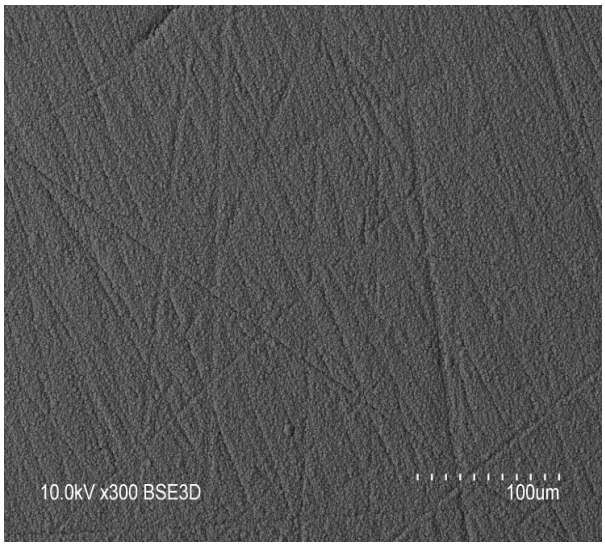

Fig. 5. Picture of the coating surface formed using aluminum oxide ceramics.

Fig. 5 shows the surface of the coating formed from aluminum oxide ceramics.

The X-ray fluorescence analysis showed the presence in the coating of all the elements that contained in the ceramic target being evaporated. It can be seen that the coating has a substantially homogeneous surface without any prominent pores; nevertheless, there are cracks present all over the surface. The absence of pores and the surface homogeneity are due to high migration ability of the adsorptive atoms [**7**] at significant substrate temperatures (over 700 ^{o}C). This fact, on the other hand, may have negatively affected the coating formation and resulted in the appearnce of micro-cracking. The substrate hight temperature caused by the irradiative heating and the obtained morphology of the coating surface may testify to the crystalline structure of the coating with the presence of Al_2O_3 γ and α phases [**7**]. However, this was not within the scope of the present studies and is a subject of our further investigations.

Conclusion

The results of the conducted investigations have demonstrated the possibility of evaporation of aluminum oxide ceramic by a fore-vacuum electron source and its use in forming coatings. Ion mass-to-charge composition of beam plasma have shown that the ions of target appear in plasma after the beginning of evaporation. The obtained deposition rate of ceramic coating (0.3 μm/min) speaks for the efficiency of the technique and its applicability to various technological processes. Alumina ceramic coating deposited on the steel sample from such multi-component beam plasma has uniform surface without pores and other damages. Further investigations will be directed toward the study of adhesion and tribological properties of this coating.

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Author's contributions

Conceived the plan: EM; Performed the expeirments: AV, YuG; Data analysis: YuG, DB; Wrote the paper: YuG, DB. Authors have no competing financial interests.

Supporting information

Supporting informations are available from VBRI Press.

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