Fore-vacuum plasma-assisted deposition of dielectric coatings and process characterization

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Abstract. The paper aims at imparting electrical insulating properties to conductor surface via deposition of dielectric coatings. The originality of the method consists in the deposition of ceramic coatings as a result of direct electron-beam evaporation of solid dielectric (ceramic) target in the fore-vacuum pressure range (1-100 Pa). As the evaporating material we used alumina ceramics. The spatial distribution (geometrical shape of vapor clouds) of ceramics vapors was also studied using the system of the identical substrates placed at different angles and distances from the target. Raster microscopy have demonstrated that we achieved uniform alumina ceramic coatings with thickness up to 5 µm (measured by a non-contact profile meter), with deposition rate of \( \approx 0.3 \) µm/min and surface resistance up to 140 MOhm (measured by therma ohmmeter).

1. Introduction

Plastics, ceramics and ceramoplastics normally provide electrical insulation in electronics [1-3]. Among the others, plastics correspond to low temperature requirements of electronic devices, but most of them do not serve for long-term time at temperatures above 300°C [4-6]. In aerospace technologies and instrument engineering [7-9], ceramic and coatings on its basis can improve the surface properties [10-12] and provide efficient heat removal from thermally loaded elements but its mechanical treatment is difficult, and the manufacturing of ceramic insulators itself is rather complicated.

Now, several modern methods are available for creating high-quality insulating coatings on conductors. For example, such coatings on metals can be deposited by micro-arc oxidation [13]. In this method, Al- or Zr-based substrate is placed in acid-containing liquid under applied voltage to initiate a micro-arc discharge. During this discharge, the metal surface is oxidized, and a ceramic coating (Al or Zr oxide) is formed on the surface. The basic shortcoming of the method is that the discharge ceases as soon as the surface becomes dielectric, i.e., once the metal surface stops drawing the discharge current, the formation of its dielectric coating ends.

Ceramic coatings can be deposited by magnetron sputtering [14], which is widely used for the formation of functional coverings. However, during the operation in active gases (nitrogen, oxygen), the target surface is covered with oxide or nitride film, that degrades discharge parameters and limits coating growth rate to tens of nanometers per minute. Therefore, such coatings are used only in electronic devices exploited at a voltage of several volts. Low growth rate of dielectric coatings is provided by plasma chemical vapour deposition [15] used in microelectronics. Nevertheless, listed methods are promising for industrial applications, and their efficiency has been constantly improved in the last two decades.
In the paper, we study the possibilities of depositing an electrical insulating coating on metals through electron beam evaporation [16] of solid ceramic and its condensation in fore-vacuum [17]. The proposed method is simple, provides rather high deposition rates (about several tens of micrometers per hour), and allows controlling the deposition parameters over a wide range [18].

2. Experimental part

In our experiments, we used a fore-vacuum electron source based on a hollow-cathode glow discharge which operated in continuous mode. The parameters of the setup and the formation of coatings are described in detail elsewhere [17]. Figure 1 shows a simplified schematic of the experimental arrangement. The fore-vacuum source produced electron beam 1 with a current of 100 mA and energy of 15 keV. The beam was focused to a diameter of 4–6 mm by the magnetic system of the source and was transported to ceramic target 3 through the space of its vacuum chamber filled with air to a pressure of 2–10 Pa, creating dense beam plasma 4.

![Figure 1. Experimental arrangement: 1 – electron beam, 2 – metal substrate, 3 – ceramic target, 4 – beam plasma.](image)

When exposed to the electron beam, ceramic target 3 evaporated with partial ionization of its vapors. The charge on the ceramic surface was neutralized by ions arriving at the surface from the beam plasma. The material evaporated from the target surface was deposited on substrate 2. The substrate material was titanium VT1-0 (with Ti percentage of 99.24% – 99.7%). In our study, we measured the ceramic evaporation rate and the coating growth rate at different beam currents and energies. The ceramic target was weighed on a high-precision scale Mettler Toledo XS204 before and after sputtering, and knowing the time, beam power, and difference in weight, we could estimate its evaporation rate. The coating thickness was determined using a Calotest CAT-S-0000 device (Switzerland). The coatings were tested for electrical insulation using an E6-13A terra-ohmmeter. Photos of the coatings surface were obtained using scanning electron microscope Hitachi TM-1000.

The spatial distribution of ceramic vapors was analyzed on an experimental test bench (figure 2) which comprised four half-arcs of different diameters with fixed substrates and a graphite crucible with ceramic at the system center.
Figure 2. Experimental test bench for analysing spatial distributions of ceramic vapours.

Test bench given in figure 2 allowed us to measure the coatings thickness which was dependent on the position of the given sample with respect to the electron beam and the focal point of the beam on the surface of the target.

3. Results and discussion

Figure 3 shows the ceramic evaporation and coating growth rates as a function of the beam current and energy. We found that the material from the ceramic target evaporated with rate of up to \( \approx 70 \text{ mg/min} \) (\( \approx 4 \text{ g/h} \)), and its deposition rate on the substrate was \( \approx 0.3 \text{ µm/min} \).

Figure 3. Ceramic evaporation rate and coating thickness vs. electron beam parameters.

Mass-charge state of ions in the beam plasma, studied in our previous work using a modified RGA-100 quadruple mass analyser, have demonstrated that a secondary plasma consisting of residual gas products and evaporated target elements was formed during electron beam evaporation [19]. Figure 4 shows the thickness of the ceramic coating on the different samples, placed at different distances and angles from electron beam. The decrease in the coating thickness with increasing the target–substrate distance is likely due to ceramic vapour scattering at gas neutrals. It should be noted
that the evaporated ceramic material moves mainly along the beam axis and its amount in the radial direction is decreased.

![Figure 4](image-url)

**Figure 4.** Coating thickness versus distance from evaporated ceramic to substrate.

Figure 4 shows that difference in coating thickness between 20 and 40 degrees is smaller than between 40 and 60, and even smaller for 60 and 80 degrees. This could be explained by the decrease of vapor fluxes at large angles due to the edges of the solid ceramics surrounding the liquid alumina bath.

Figure 5 shows images of thus deposited ceramic coating. As can be seen from figure 5, the coating is rather homogeneous and nonporous but cracks are present over its entire surface. The homogeneous and nonporous coating surface owes to the high mobility of adsorption atoms at substrate temperatures higher than 700 °C [20]. At the same time, high mobility of atoms as well as high temperature can be also responsible for micro-cracks. High substrate temperature coupled with observed surface morphology suggests that the coating has a crystalline structure containing γ- and α-Al₂O₃. However, this issue requires a separate analysis, which is expected in our further studies.

![Figure 5](image-url)

**Figure 5.** Surface of alumina ceramic with micro-cracks.
Figure 6. Surface resistance of coating vs. its thickness.

The initially-measured surface resistance of the alumina ceramic target before evaporation was $10^6$ MOhm. As can be seen from figure 6, the resistance of the coating increases linearly with increasing of its thickness, and this linearity allows one to provide a desired resistance of coatings by properly choosing their deposition mode. One can see that surface resistance of obtained coatings is in several orders of magnitude lower than the value for alumina ceramics. This could be explained as follows. During evaporation of alumina ceramics, some of the $\text{Al}_2\text{O}_3$ molecules dissipate under the beam, plus, alumina vapor additionally evaporates from the liquid phase on the surface of the target. So, the coating may contain atoms of conducting aluminum, which reduces surface resistance. Further experiments require improving surface resistivity of the coatings.

4. Conclusion

Our research demonstrates the capability of fore-vacuum plasma electron sources to provide the formation of electrical insulating coatings based on alumina ceramics. The evaporation rate of ceramics and the deposition rate of its vapours increase with increasing the beam power density. The vapours move mainly normal to the surface, and the coating growth rate decreases with increasing the distance between the substrate and sputtered target. The surface resistance of the coating increases linearly with increasing its thickness. Vapour cloud research has shown that uniform coatings with similar deposition rates can be formed on the substrates in case if the substrates are placed within $\pm 20\text{-}60^\circ$ with respect to the normal to the target. Increase of this angle up to 80° leads to the significant drop (several times) in deposition rates; however, even at such sharp angles, deposition is still possible. These findings show that within one experimental trial, our method allows depositing uniform ceramic coatings with similar deposition rates on numerous substrates placed around the target at different mentioned angles. Further research will be dedicated to study of thermal fields on these substrates placed at different distances and angles, in order to find the deposition regimes for thermal-sensitive substrates.

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