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Gas-metal e-beam-produced plasma for oxide coating deposition at fore-vacuum pressures

This article describes an experiment on the deposition of oxide coatings on silicon and metal substrates from a gas-metal plasma. The plasma was produced by e-beam evaporation of metals (Mg, Al) with subsequent ionization of gas (oxygen) and evaporated material particles at fore-vacuum (1–10 Pa) pressures. We studied the ion composition and species fraction in the gas-metal plasma (using quadruple mass-spectrometry), as well as the thickness and surface resistivity of the coatings.

Keywords: fore-vacuum, plasma-cathode electron source, coating deposition.

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A gas-metal plasma is a plasma containing ions of both gaseous and metallic species with controlled relative content. Such plasmas are of paramount interest for the deposition of nitride [1] and oxide [2] films and complex composite structures [3] on the surfaces of chosen substrates. Gas-metal plasmas for technological applications are routinely produced using arc discharges with added gas flow, or a magnetron discharge in self-sputtering mode. In recent work we have demonstrated the advantages of using a fore-vacuum pressure, plasma-cathode, electron beam source [4–7] for various applications including the generation of gas-metal plasmas by electron beam evaporation of metals in oxygen for the deposition of oxide coatings [8]. Here we describe our further research on the features of gas-metal plasmas at fore-vacuum pressures and the properties of deposited coatings.

Experimental

The experimental setup is shown in Fig. 1. The vacuum chamber was pumped using only a fore-vacuum rotary vane pump (Edwards 80) to a base pressure of approximately 1 Pa. Oxygen or helium gas was introduced into the chamber, and the working pressure was set up to 10 Pa.

The electron beam was focused by a short magnetic lens to a diameter of around 5 mm. The beam current was measured using a collector placed into the beam without the target. The electron beam focal point was located on the surface of a metal (Al or Mg) target placed under the beam on a graphite crucible. The beam heated, melted and evaporated the target, and ionized the background gas and evaporated particles, thus producing a gas-metal plasma in the region adjacent to the substrate. This plasma was used for deposition of the coating on a thin substrate made of various materials (stainless steel, corundum ceramics, or silicon) and of area $1.5 \times 1.5 \text{ cm}^2$. The substrate was located 4 cm from the e-beam axis and 3 cm above the target surface perpendicular to it. The deposition duration was measured from the visually observed beginning of target melting, and was varied from 1 to 8 min. The energy of ions bombarding the substrate was controlled by a bias voltage of up to 30 V supplied to the bias electrode of 8 cm diameter positioned under the crucible. Mass-to-charge composition of the gas-metal plasma was monitored by

a modified quadruple mass-spectrometer RGA-100. The entrance aperture of the spectrometer was located opposite the substrate and 3 cm from the beam axis; its volume was pumped by a turbo pump to a base pressure of less than 0.01 Pa. Micrographs of the deposited coatings as well as the elemental composition were acquired with a Hitachi TM-1000 scanning electron microscope (SEM). The coating thickness was measured using a Calotest CAT-S-0000. The coating profile was investigated using a 3D non-contact profilometer Micro Measure 3D Station. For coating surface resistivity measurements, two concentric copper electrodes were formed on the surface of the films. These electrodes were deposited by e-beam evaporation of copper in a helium atmosphere at a pressure of 2–3 Pa, in the same experimental setup. The resistance (Ohms) of the oxide coating between the two copper electrodes was measured by a tera-ohm meter E6-13A, and the surface resistivity ($\text{Ohm}/\square$) and specific resistivity ($\text{Ohm}\cdot\text{m}$) was calculated using well-known methods taking into account the copper electrode area and coating thickness.

Fig. 1. Experimental setup [8]: 1 – vacuum chamber; 2 – plasma-cathode electron source; 3 – electron beam; 4 – focusing system; 5 – metal (Al or Mg) target; 6 – graphite crucible; 7 – gas-metal beam plasma; 8 – substrate; 9 – substrate holder; 10 – bias electrode; 11 – modified quadruple ion mass analyzer RGA-100 [9]

Results and discussion

The mass-to-charge spectra of ions extracted from the gas-metal plasma show that, under similar experi-

mental conditions, the fractional content of metal ions depends on the kind of metal evaporated by the beam (Fig. 2).

Fig. 2. Fraction of gas and metal ions in the plasma during evaporation of Al (upper) and Mg (lower), in oxygen at 3.5 Pa. For both cases, beam current was 25 mA, beam energy 7 keV

One can see that for the same e-beam power density the fraction of Mg ions in plasma is greater than for Al. We explain this as due to the physical properties of aluminum such as boiling point and specific heat (2792 K and 284.1 kJ/mole) being more than twice those for magnesium (1363 K and 131.8 kJ/mole). Thus the aluminum evaporation process and the fraction of its ions in the plasma are significantly less for the same experimental parameters.

The surface resistivity of the coating deposited on corundum ceramic from a magnesium-oxygen plasma for a 1 min deposition period is higher than the surface resistivity of coating deposited on a steel substrate (Table 1).

Table 1

Surface resistivity for Mg oxide coatings

Substrate material	Surface resistivity, MOhm/□	Experimental conditions
Stainless steel	1.2–3.5	Beam current 75 mA and energy 6 keV, $t = 1$ min
Corundum ceramics	3.5–12	

The specific resistivity and the thickness of the coating deposited from aluminum-oxygen plasma increases with time (see Fig. 3).

SEM photos of aluminum-oxide coatings deposited on a stainless steel substrate with a copper mask are

shown in Fig. 4, displaying the copper mask as well as the thickness measuring method.

SEM photos of the oxide coatings deposited on a silicon substrate are shown in Fig. 5, revealing the high degree of uniformity of these coatings.

Fig. 3 Coating thickness (left axis) and specific resistivity (right axis) vs. time of e-beam evaporation of an Al target in oxygen. Experimental conditions: beam current 95 mA, beam energy 13 keV, oxygen pressure 10 Pa

Fig. 4. Aluminum-oxide coating on stainless steel, with copper mask, for deposition times 2 min (left) and 4 min (right). Experimental conditions: beam current 95 mA, beam energy 13 keV, oxygen pressure 10 Pa

Fig. 5. Photos of the surfaces of coatings deposited on Si substrates from plasmas of oxygen and metal – Mg (left) and Al (right)

Conclusion

An electron-beam produced gas-metal beam plasma has been utilized for the deposition of oxide coatings on steel, silicon and corundum ceramic substrates. We note that the specific resistivity of aluminum-oxide films formed by this method has a value which is between the resistivity of anodized Al_2O_3 (dielectric, with specific resistivity of $\sim 8 \cdot 10^8$ $Ohm \cdot m$) as well as pure aluminum (metal, $2,6 \cdot 10^{-8}$ $Ohm \cdot m$), and differs by at least eight orders of magnitude from these limit points.

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Газо-металлическая электронно-пучковая плазма для осаждения оксидных покрытий в форвакуумной области давлений

Статья описывает эксперимент по осаждению оксидных покрытий на кремниевые или металлические образцы из газо-металлической плазмы. Такая плазма генерировалась при электронно-лучевом испарении металлов (Mg, Al) с одновременной ионизацией газа (кислород) и частиц испаренного вещества в форвакуумном диапазоне давлений (1–10 Па). Исследованы: ионный состав плазмы, доля газовой и металлической ионной компоненты (с использованием квадрупольного масс-спектрометра), толщина и поверхностное сопротивление покрытий.

Ключевые слова: форвакуум, электронный источник с плазменным катодом, осаждение покрытий.