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Abnormal glow discharge in crossed electric and magnetic fields in the presence of reactive gas

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Abstract. The results of investigations of the low-temperature plasma at process of reactive magnetron sputtering of Ti target in the presence of reactive gas are described. Discharge volt-ampere characteristics for different schemas of reactive gas input are build. Optimal regimes of making strengthening coatings are defined. TiO_x ($0<x<2$) strengthening coatings at plastics are made. Coatings showed high consumer qualities.

1. Introduction

Low-temperature plasma is widely used for making functional coatings. One of the most popular coating methods is magnetron sputtering [1-3]. This method is applying for making different multilayer functional coatings (strengthening, low emission, antireflection, etc.) at wide range of materials: glass, plastic, ceramic, metal [4]. Their structure could consider oxide layers. Such oxide layers could be coated by magnetron sputtering of metallic target in the presence of reactive gas – oxygen. Process of coating at plastic substrate is difficult because of low plastic melding temperature, consequently temperature regime of coating should be chosen correctly [5].

The aim is to make strengthening coatings on plastic by low temperature plasma of magnetron discharge.

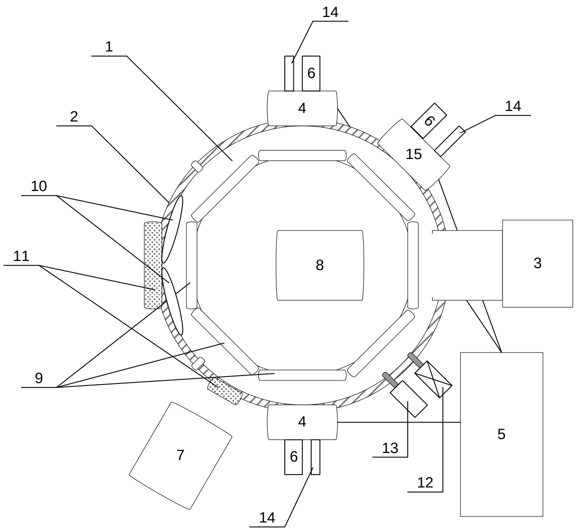
2. Experimental plant and methods

Functional schema of experimental plant is showed at pic. 1. It consists of: vacuum chamber 1, door 2, pump system 3, two magnetrons 4, power supply of magnetron and source of ions 5, system of gas input, system of photometric control of film thickness that include spectrophometer S-100, moving system 8 of substrates 9, heating system 10, windows 11, valve for atmosphere air 12, filled-system transducer 13, cooling system 14, ion source 15 [6] .

There is control stand for all systems. Pumping system includes mechanical pump 2NVR90B and diffusion pump ND-200 and provides vacuum at chamber about 10^{-3} Pa. Pressure registration system consists of vacuum-gauges VT1-4 (thermocouple method) and VMB1-2 (ionization method). Gas input in chamber realized by leak valves VTT02.01.31.000 and controller RRG-8. Gas flow counted by constant volume method [7]. Spatial distribution of temperature in region of magnetron measured by probe method at different distances from magnetron.

Volt-ampere characteristics measured within input of work and reactive gases in vacuum chamber at different ratio. Voltage fixed at different values of discharge current. Experimental plant provides gas input in region of magnetron target (schema “A”) and in region of substrate (schema “B”).

In case of schema "A" reactive gas atoms concentration increases near target surface and probability of their condensation at target is more. In case of schema "B" oxygen atoms concentration near target magnetron is low, consequently, probability of their condensation at target is lower. Oxide appears at target surface because of its high temperature that needs for chemical reaction of oxidation. Target surface oxidation depends on quantity of adsorbed reactive gas atoms, so in case of schema "A" the process is more intensive.



Pic. 1. Functional schema of experimental plant: vacuum chamber 1, door 2, pump system 3, two magnetrons 4, power supply of magnetron and source of ions 5, system of gas input, system of photometric control of film thickness that include spectrophometer S-100, moving system 8 of substrates 9, heating system 10, windows 11, valve for atmosphere air 12, filled-system transducer 13, cooling system 14, ion source 15.

Discharge parameters: cathode voltage 350 V; discharge current 8 A; vacuum in chamber 0,1-0,4 Pa. Target material – Ti. Work gas (Argon) flow was 0,4 – 1,2 mg/sec, reactive gas (Oxygen) flow was 0,2 – 0,7 mg/sec. Sputtering time was 10-30 minutes. Substrates: polycarbonate (PC) and polyethylene-terephthalate (PET). Substrate thickness: 3,0 and 0,45 mm accordingly. As witness was optical glass K-8. There was no additional heating of substrates. Before coating substrates were cleaned by ions from ion source [8]. Film thickness was from 50 to 150 nm.

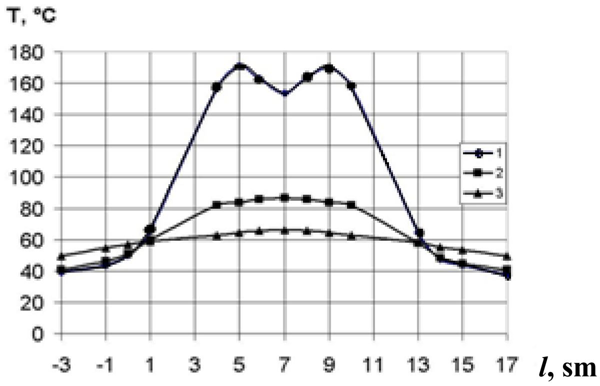
Titanium oxide films at polycarbonate substrates strength characteristics were measured by micro durometer PMT-3M. Titanium oxide films at polycarbonate substrates strength characteristics were measured by SM-55 according to OST 3-1903-95 [9]. Also two-dimensional method for integral characteristics measuring was used [10]. This method defines film's mechanical characteristics in system "coating-substrate" [11].

3. Results and discussions

Temperature spatial distribution in vacuum chamber at region of magnetron is shown at pic. 2. For making coatings on plastics optimal distance is 10 sm, because of low and equal heating and no film destruction. At the same time maximal coating speed is provided.

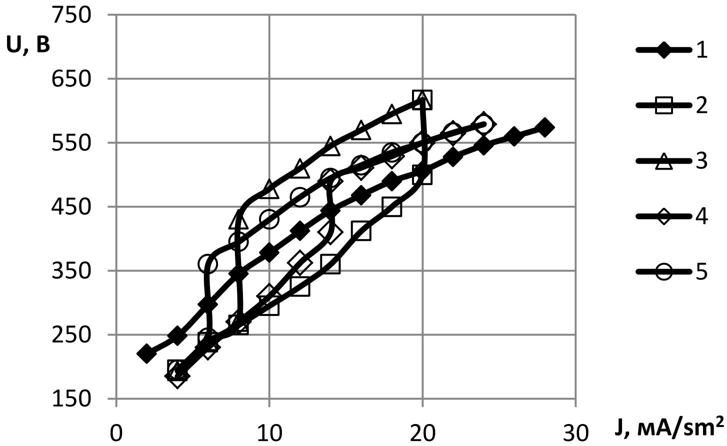
At reactive magnetron sputtering of metallic target with direct current reactive gas addition leads to changes of discharge volt-ampere characteristics. There are volt-ampere characteristics at pic. 3 for sputtering only in Argon and in mixture of Argon and Oxygen at different gas input schemas.

Oxide film at target surface appears at work and reactive gas mixture input in chamber. When discharge is burning the transparent coating is making. Increasing of discharge power at defined moment oxide film at target surface sputters totally and metallic coating is making. Discharge power decreasing results to opposite effect but at lower value. The same hysteresis was showed in [12].



Pic. 2. Temperature spatial distribution in vacuum chamber at region of magnetron. 1 – distance *l* between magnetron target and substrate is 6 sm, 2 – 12 sm, 3 – 18 sm.

At this graphics lower part of hysteresis describes sputtering of oxide film from target surface and making transparent coating on substrate. Higher part of hysteresis describes sputtering of metal target and making metallic coating on substrate.

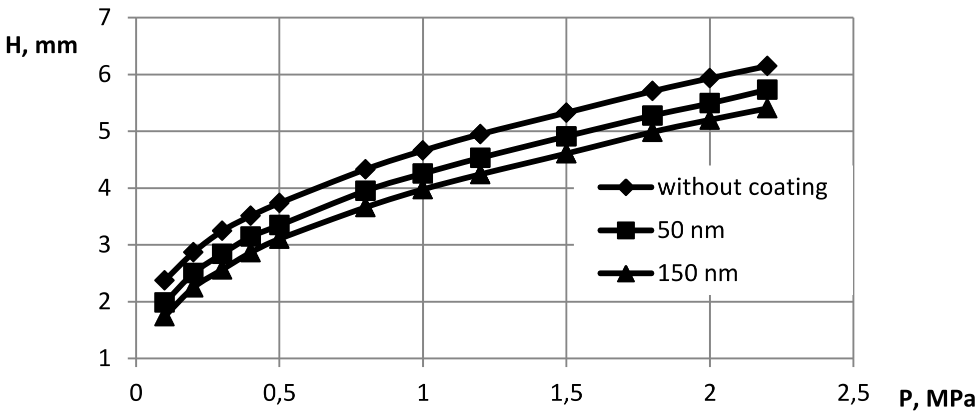


Pic. 3. Volt-ampere characteristics for different work and reactive gas schemas: 1 – pure Argon, 2,3 – Argon and Oxygen mixture schema “1”, discharge power increasing (2) and decreasing (3), 4,5 – Argon and Oxygen mixture schema “2”, discharge power increasing (4) and decreasing (5)

Oxidation degree of coating at substrate depends on ratio or work and reactive gas in vacuum chamber. Therefore, choosing gas input schema and controlling gas ratio it is possible to manage oxidation degree, which influences on optical and strength characteristics of coating. This method differs from analogical methods described in [13].

Micro hardness (HV) of coatings on PC substrates achieved about 30 kg/mm^2, that is equal to micro hardness of substrate. Coatings of titanium oxide showed 3-rd hardness group according to OST 3-1903-95. Hence this coatings can be used for strengthening of plastic and defend its surface from mechanical wear.

PET substrates with and without coatings mechanical characteristics are showed at pic. 4. Coating that has thickness 50 nm effects on flexure of sample. Thickness increasing enlarges effect. Flexure decreasing means that system “substrate-coating” became harder.



Pic. 4. Experimental flexure H of samples depending on pressure P.

4. Conclusions

Volt-ampere characteristics for different schemes of introduction reactionary and working gases are built. A new method for controlling the level of oxidation coating is proposed. Optimal regime of making oxide coatings on a plastic substrate, taking into consideration the input of gases and low melting temperature of the substrate material, is selected. Micro-hardness of coatings on PC and PET is about 30 kg/mm^2. PET substrates coated TiO_x ($0<x<2$), which thickness up to 150 nm, have 10-30% higher stiffness than PET film without coating. Coatings TiO_x correspond to the 3rd group of abrasion resistance according to OST 3-1901-95 and can be used for surface hardening of plastic. Mechanical characteristics of titanium oxide films provides using this films for strengthening and defending from mechanical wear plastic surface.

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