

EROSION OF VACUUM-ARC TiN COATINGS IN PLASMA OF STATIONARY MAGNETRON-TYPE DISCHARGES

G.P. Glazunov, A.A. Andreev, M.N. Bondarenko, A.L. Konotopskiy, V.A. Stolbovoy

National Science Center “Kharkov Institute of Physics and Technology”, Kharkov, Ukraine
E-mail: glazunov@ipp.kharkov.ua

Studies were made into erosion of titanium nitride coatings deposited onto stainless steel substrates in two different ways of vacuum-arc technology: with and without feeding high-voltage negative pulses on the substrate. A possible physical mechanism is offered and discussed to explain the different character of erosion behavior.

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1. INTRODUCTION

Inasmuch as protective TiN coatings are used in the manufacture of various components of magnetic plasma confinement facilities Uragan-2M and Uragan-3M (IPP NSC KIPT, Kharkov), it is necessary to know their vacuum-plasma characteristics such as the erosion rate in the interaction with fast particles, outgassing and sorption in vacuum, hydrogen permeability, etc. As regards the erosion rate, it has been previously found [1-3] that TiN coatings have a doubtless advantage over stainless steel, the sputtering rate of which caused by hydrogen ions is approximately three times higher. However, those data have been obtained at room temperature of the samples. The literature data on the erosion rate at high temperatures and at exposure to nitrogen plasma are absent. At the same time, during operation of TiN-coated units (limiters, RF antenna components, etc.) supposed to be used in the Uragan-2M torsatron, the units will be exposed to powerful plasma flows, including periods of cleaning by steady-state discharges in argon, nitrogen, helium, hydrogen, atmospheres. The working temperature of coatings in this case may be substantially higher than room temperature. Therefore, it has appeared expedient to investigate the erosion behavior of TiN coatings at high temperatures under the action of plasma. Besides, considering that recently a new technique has appeared for vacuum-arc TiN coating deposition through supplying high-voltage (HV) pulses to the substrate during sputtering [4], it became of interest to compare the erosion characteristics of the coatings of two types between themselves and with the ones of stainless steel.

2. EXPERIMENTAL AND RESULTS

The experiments were carried out at the DCM-1 device (bench for diagnostic of materials under plasmas irradiation) under operating conditions of magnetron-type cylindrical-symmetry discharge [5]. The typical discharge parameters were as follows: magnetic field in the region of discharge ~ 0.05 T, working gas pressure 0.2 Pa, discharge voltage 0.4 to 1 kV, the discharge current varied within 60 to 180 mA. The Langmuir probe was used to measure the plasma column edge characteristics such as electron temperature T_e , electron density n_e , plasma potential ϕ .

The typical edge plasma characteristics of magnetron-type discharges in nitrogen atmosphere were: $T_e \sim 0.2...0.3 \times 10^4$ K, $n_e \sim 2...3 \times 10^9$ cm $^{-3}$, $\phi \sim 7$ V. The samples presented $200 \times 10 \times 0.3$ mm straps from stainless steel 12Kh18N10T, on both sides of which $10...20$ μ m thick titanium nitride coatings were deposited by two different technologies: i) with feeding HV negative potential pulses to the substrate [3], and ii) without feeding the mentioned pulses (traditional technology [6]). The technique of TiN coating deposition has been described in sufficient detail in ref. [7]). The erosion rate s ($\text{r/s}\cdot\text{cm}^2$) or the sputtering coefficient α (atom/ion) was the measured parameter. Before measurements, the samples were annealed for half an hour at temperatures between 800 and 900°C in a special vacuum chamber at a pressure of 5×10^{-7} Torr, and then were fixed as a cathode in the DCM-1 facility. The sample temperature during irradiation was assigned by the ion current value and ranged from 500 to 1000°C. The erosion rate was determined by the weight loss method with analytical balance weighing of samples before and after plasma treatment of the coatings, and was calculated by the equation: $\alpha = (p_1 - p_2) / 6.25 \cdot I \cdot t \cdot m$, where p_1 and p_2 denote the sample weight before and after irradiation, respectively; I is the ion current (A), t is the process time (s), m is the average atomic weight of the cathode material (g). Figs. 1 and 2 show erosion coefficient (rate) as a function of the ion current (temperature) for TiN coatings of two types and stainless steel, expressed in different units – atom/ion and $\text{g/s}\cdot\text{cm}^2$.

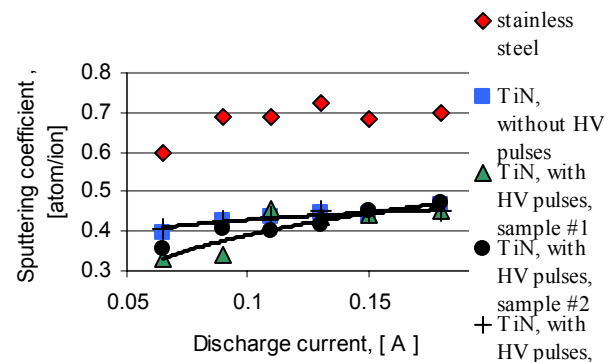


Fig. 1. Sputtering coefficient of TiN coatings and stainless steel versus discharge current

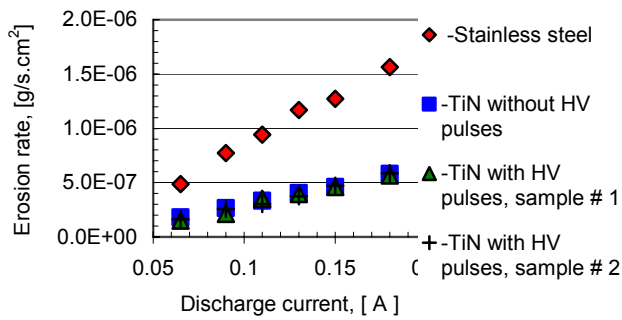


Fig. 2. Erosion rate ($\text{g/s}\cdot\text{cm}^2$) of TiN coatings and stainless steel versus discharge current

The dependencies on discharge current for various bombarding ions (hydrogen, helium, argon) are presented in Fig. 3.

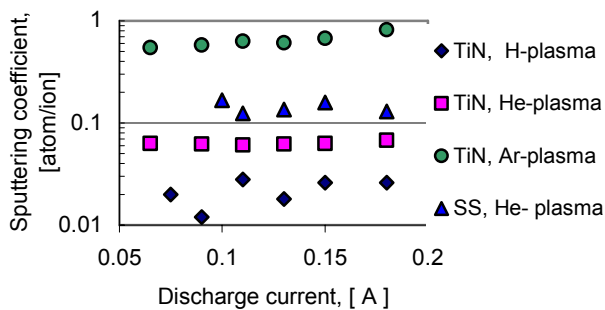


Fig. 3. TiN coating sputtering coefficient versus current values of discharges in hydrogen, helium and argon

The measured dependence of erosion on the atomic number of bombarding ions is shown in Fig. 4. Its character is similar to that measured earlier for tungsten [8].

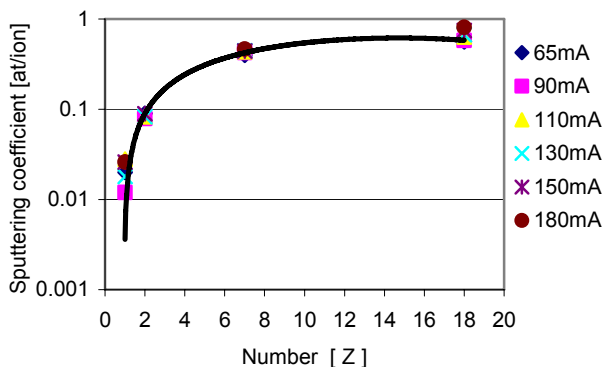


Fig. 4. TiN-coating sputtering coefficient for various discharge current versus bombarding ion atomic number Z

3. DISCUSSION

It is obvious from Figs. 1 and 2 that at high temperatures the absolute value of erosion rate for the TiN-coated samples is also essentially (about two times)

lower than that for the uncoated stainless steel samples, as it has been previously observed [1] at room temperature. Notice that it is true for the samples having TiN coatings deposited by different technologies, i.e., with and without supply of high-voltage pulses. This essential reduction in erosion of titanium nitride as opposed to stainless steel or titanium may be attributed to the effect of preferential sputtering of a lighter component, namely, nitrogen. So, in paper [2] it has been demonstrated that as early as at hydrogen ion irradiation doses of $3 \times 10^{19} \text{ cm}^{-2}$ the subsurface layer of the TiN film consists mainly of titanium.

In the $500 \dots 1000^\circ\text{C}$ temperature range under study, the sputtering (erosion) coefficient expressed in atom/ion units is weakly dependent on the sample temperature, this being in agreement with the earlier obtained data on sputtering of vacuum-arc tungsten coatings [5]. This is explained by high melting temperatures of the materials investigated. However, if the measured data are represented in terms of the erosion rate ($\text{g/s}\cdot\text{cm}^2$), then the latter increases proportionally with the discharge current. At the initial stage of irradiation in nitrogen plasma (in Fig. 1, this corresponds to low discharge currents), the sputtering coefficient of TiN films deposited with feeding HV pulses to the substrate is 20 to 30% lower than that for the samples prepared by the traditional technology (without supply of HV pulses to the substrate). However, with an increase in the irradiation dose the erosion also increases and becomes identical for the both types of the coatings. This result can be explained as follows. In the experiments made previously in ref. [7] it has been revealed that for the vacuum-arc TiN coatings there exist two regions of outgassing with different nitrogen-binding energies at 600°C and 800 to 900°C . At that, the coatings obtained by the traditional technology (without pulse supply) release more nitrogen in the first region, i.e., at 600°C , while at higher temperatures more nitrogen is released from the coatings obtained under the conditions with high-voltage pulses. It has been assumed [7] that the HV pulse supply regime is favorable for an intense capture of a greater amount of nitrogen with an enhanced binding energy due to the presence of an additional source of ionization. If it is so, then at the initial stage of irradiation the mentioned coatings are more erosion resistant in the sense that instead of titanium more nitrogen is sputtered. As the irradiation dose increases, owing to a selective sputtering of nitrogen, the stoichiometric composition of the surface layer of the coating changes (gets enriched with titanium) and the erosion rates of the two types of coatings are practically not different. In conclusion, we note that at a repeat determination of the erosion rate on the samples exposed to high ($> 10^{20} \text{ cm}^{-2}$) irradiation doses, the above-mentioned effect is not observed at low irradiation currents (see Fig. 1, curve +).

The erosion dependencies on discharge current in the case when TiN coatings were irradiated in plasmas of steady state discharges in other gases atmosphere, namely: in hydrogen, helium and argon, have the same character as for nitrogen plasma (Fig. 3). I.e., sputtering coefficients weakly depend on discharge current value, and its absolute value is essentially lower than that for stainless steel.

4. CONCLUSIONS

Under irradiation with steady state nitrogen plasma in the sample temperature range from 600 to 1000°C the erosion rates of vacuum-arc TiN coatings produced by different technologies (with and without feeding high-voltage negative pulses to the substrate) are approximately by a factor of 2 lower than those of stainless steel. At the initial stage of irradiation, the erosion coefficient of TiN films deposited with feeding high-voltage pulses to the substrate is 20 to 30% lower than that for the samples prepared by the traditional technology (without supply of high-voltage pulses to the substrate). Perhaps, this may be caused by an increased content of nitrogen with a higher binding energy in the coatings deposited with supply of high-voltage pulses to the substrate. As the irradiation dose increases, the erosion rates of the two types of coatings are practically not different, this being most probably due to the change in the stoichiometric composition of the TiN coating surface caused by a selective sputtering of nitrogen.

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ЭРОЗИЯ ВАКУУМНО-ДУГОВЫХ TiN-ПОКРЫТИЙ В ПЛАЗМЕ СТАЦИОНАРНЫХ РАЗРЯДОВ МАГНЕТРОННОГО ТИПА

Г.П. Глазунов, А.А. Андреев, М.Н. Бондаренко, А.Л. Конопский, В.А. Столбовой

Исследована эрозия покрытий из нитрида титана, осажденных на подложки из нержавеющей стали двумя различными методами вакуумно-дуговой технологии: с подачей и без подачи высоковольтных импульсов отрицательной полярности на подложку. Предлагается и обсуждается физический механизм для объяснения различного характера эрозионного поведения.

ЕРОЗІЯ ВАКУУМНО-ДУГОВИХ TiN-ПОКРИТТІВ В ПЛАЗМІ СТАЦІОНАРНИХ РОЗРЯДІВ МАГНЕТРОННОГО ТИПУ

Г.П. Глазунов, А.А. Андреев, М.М. Бондаренко, О.Л. Конопський, В.А. Столбовий

Досліджено ерозію покриттів з нітриду титану, осаджених на підкладки з неіржавіючої сталі двома різними методами вакуумно-дугової технології: з подачею і без подачі високовольтних імпульсів негативної полярності на підкладку. Пропонується і обговорюється фізичний механізм для пояснення різного характеру ерозійної поведінки.