

# EROSION, PERMEATION AND OUTGASSING PERFORMANCES OF TiN COATING UNDER/AFTER HYDROGEN PLASMA IRRADIATION

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An erosion behavior of TiN-coated stainless-steel (SS) surfaces was investigated during biased-limiter experiments within the Uragan-3M torsatron and during simulation experiments, which were performed with plasma-accelerator and glow-discharge (GD) plasmas. For a TiN-coated SS head-plate of a limiter the arc ignition probability was found to be lower than  $10^{-4}$  per plasma pulse. Possible physical mechanisms of this effect had been discussed. Within special vacuum stands, using thermal-desorption and mass-spectrometry methods, there were performed measurements of an outgassing rate and hydrogen permeability of TiN-coatings. The negligible outgassing from TiN-coated SS samples, during their heating up to 473 K, was observed after a cleaning procedure with a molecular hydrogen inflow under pressure of about  $10^{-4}$  Torr, on contrary to the considerable increase of (q) rate for the irradiated samples. Measured values of the TiN-film hydrogen permeability were several times lower, and activation energy of the hydrogen permeation was considerably lower than that for the SS films (15 kJ/mole instead of 19.9 kJ/mole). The use of TiN-coated SS and diffusion membranes, for the reduction of the erosion, recycling, and hydrogen isotope inventory control, as well as for improvement of vacuum conditions, has been considered.

## 1. Introduction

The modification of SS components by titanium-nitride (TiN) coatings has been widely used in technology of the manufacturing of RF antennas, shields, movable and unmovable limiters for the Uragan-3M (U-3M) and Uragan-2M (U-2M) torsatron-type facilities. When a TiN-coated RF antenna frame was applied, a considerable reduction in the unipolar arc erosion of antenna surfaces, and a decrease in amount of heavy impurities in plasma, had been observed [1]. Also, the TiN coating sputtering coefficient, under the ion irradiation, had been found to be lower by factor of 3 as compared with that for stainless steel. For hydrogen discharges within U-3M, however, some uncontrollable disruptions of the TiN-coating (upon the SS shields) were observed after two experimental campaigns. Considerable changes in coating colors, from golden for non-irradiated parts of shield - to silvery, orange, red, and dark-blue color for heavily irradiated parts, were observed. The data obtained in previous studies [2], with help of the Auger electron spectroscopy (AES), X-ray spectroscopy (XRD), fluorescence (EDX) diagnostic methods, allow to suggest that these color changes could be caused by various alterations in the stoichiometric composition of a TiN-film surface layer after the interaction with U-3M hydrogen plasma. It must be noted, that such damages were not observed after the hydrogen-ion irradiation of the same TiN coatings, as described in Ref. [3], but a considerable Ti enrichment of the film surface layer was found. On the contrary, the film color changes took place after the high-power pulsed hydrogen-plasma irradiation at certain energy densities and fluencies [2]. The TiN erosion values, as measured in this work, were considerably higher than those observed for bare SS. The nature of the disruptions mentioned above and mechanisms of such an erosion behavior of the TiN-coating were not explained enough. Taking into account, that large-surface TiN-coated SS shields and RF-antennas, which were installed in U-2M, have to be operated in future, additional investigations of a TiN-coating behavior under the pulsed plasma irradiation are

needed. Besides that, in order to get a data base for control of vacuum conditions, erosion, hydrogen recycling, and inventory processes, there was necessity to study the outgassing and hydrogen behavior characteristics of the TiN coatings. Such information could also be useful for TiN surface modification technologies used today, and in particular for ultra-high vacuum applications [4], for an improvement of the RF-window breakdown resistance [5], etc.

## 2. Experiments and results

### 2.1. Erosion behavior in biased-limiter experiments

For the experiments with a biased limiter a new version of the movable limiter was installed within the U-3M vacuum chamber. Instead of a hot-pressed boron-carbide tile [6], the limiter head plate material was 12KH18N10T SS coated with 5- $\mu$ m-thick TiN-film, which was deposited by means of the plasma-flux condensation method [7]. The scheme of experiment was similar to that described in Ref. [6]. The limiter plate edge was located at a distance of 14 cm from the plasma column axis (at the plasma edge position). In order to investigate a TiN erosion behavior there were channels for measurements of TiII and NII spectral lines and eleven collecting probes placed on plasma facing surfaces of the helical winding housings. Tests of the limiter material were performed in the ICRF plasma discharge cleaning-regime. Typical operational parameters were as follows: hydrogen pressure  $p = 10^{-4}$  Torr,  $n_e = 2 \times 10^{12}$  cm<sup>-3</sup>,  $T_e = 10$ -15 eV,  $B = 0.035$  T, plasma pulse duration  $t = 50$  ms, pulse frequency  $f = 0.2$  Hz, total discharge power  $W = 80$  kW at frequency of the RF generator equal to 5.4 MHz. The limiter head plate was biased negatively to  $-(120$ -200) V, and the bias pulse duration was 10-50 ms. After  $10^4$  plasma cleaning discharges (no arc ignition was observed during that period) the limiter was removed from its position near plasma edge region and it was placed within a divertor slot. No increase in NII or TiII line intensity was observed for biased/unbiased regimes of the experiments, both for edge plasma and divertor plasma positions. After the exposure of the limiter to

divertor plasma during  $10^3$  work discharges, when the experimental campaign was finished, the TiN-coated head plate was dismantled for an analysis.

There were observed no damages of the TiN surface, except for weak change of the TiN-film color on that part of head plate surface, which had the direct contact with the edge plasma. Taking into account the number of plasma pulses, probability of the arc ignition for TiN was estimated to be less than  $10^{-4}$  per pulse, instead of  $10^{-1}$  measured for the previous boron-carbide limiter [8]. Also no increase in collecting probe weights was found, in contrary to the previous experiments with the B4C limiter [9]. These results indicate only a higher erosion resistance of the TiN-coated SS surfaces to the plasma irradiation, under biased-limiter experiment conditions. In order to shed some light on the nature of the TiN-coated SS shield disruption, some simulation experiments were undertaken.

## 2.2. Erosion behavior in simulation experiments

The simulation studies of an erosion behavior of TiN-coated SS samples were performed with pulsed hydrogen plasmas produced by a plasma accelerator, and with stationary glow-discharge (GD) plasmas. In the first case the specimens of 10x25 mm size, which were cut out from a non-irradiated part of the U-3M RF-antenna shield, had identical performances to the above-mentioned limiter head-plate material. It should be noted that that shield was located between the RF-antenna frame and a housing of the helical windings, and it was exposed under typical U-3M operational parameters during two experimental campaigns. It should, also, be added that the shield, after its dismantling and removing from the U-3M vacuum chamber, was exposed to atmospheric air during about 12 months. A scheme of the irradiation, operational regimes and diagnostics, were described in details in previous papers [2, 10]. Here will be described only data about the erosion dependence on an energy density and a number of pulses, in order to discuss a physical mechanism of the TiN-coating disruption. It can be seen in Fig.1a that the erosion value depends weakly on an energy flux density in spite of a different character of the disruption process. It should be noted that all points in Fig.1a present results of the irradiation with single plasma pulse. According to estimations the energy flux density decreased from  $5.5 \text{ J/cm}^2$  to  $1.4 \text{ J/cm}^2$  when a distance was changed from 30 cm to 60 cm. With such an energy density variation the net erosion value was changed from  $5 \times 10^{-4} \text{ g/cm}^2$  per pulse to  $4 \times 10^{-4} \text{ g/cm}^2$  per pulse. An optical analysis of the surface morphology (Fig 2b) had shown that the melting of a TiN-film takes place under the higher energy density (the TiN melting temperature is 3227 K). In the case of a lower energy density ( $1.4 \text{ J/cm}^2$ ) the melting did not appear (Fig.2c), but there were observed some flakes and regions of the SS substrate free of the TiN-film, due to its flaking (or shelling). Also, various changes in the film color (from orange or red to dark-blue color) were observed. Similar TiN-film color changes were observed after a GD hydrogen- and helium-plasma treatment. In that case the GD plasma performances and experimental conditions were similar to those described in Ref [11]. The

irradiation doses were about  $10^{19} \text{ ion/cm}^2$ , and an average energy value for ions was about 200 eV. The samples under studies were palladium pipes (of 0.6 cm in diameter, 0.025 cm in thickness, and 19 cm in length) coated with a 3- $\mu\text{m}$ -thick TiN layer. In that case the TiN deposition conditions were identical to those applied for the TiN-coated SS-shield or TiN-limiter. Temperatures of the samples during a plasma treatment were in different experiments equal to 623-723 K. After the irradiation with hydrogen- and helium-plasmas drastic changes in the coating color (from orange to dark blue), in dependence on a dose and temperature value, were observed visually. It should be noted that such color changes were not observed after the irradiation by GD nitrogen-plasma. Moreover, it was possible to reproduce the initial film color by an appropriate treatment with GD nitrogen plasma.

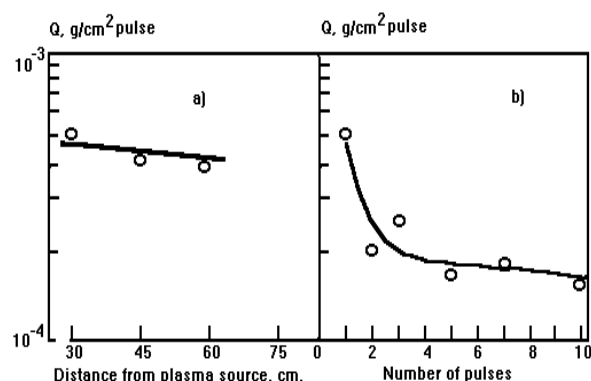


Fig. 1. The erosion value dependence of (a) distance from plasma source and (b) number of pulses

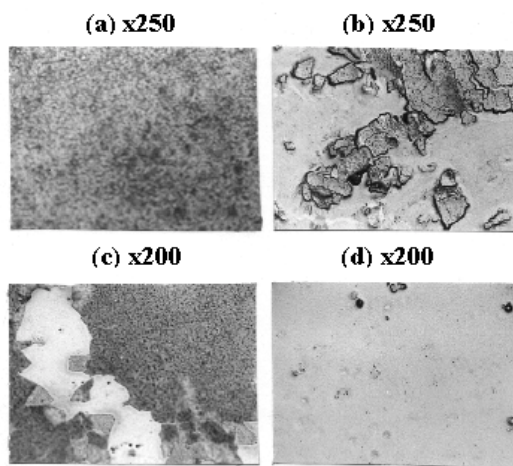


Fig. 2. TiN-coating surface for (a) non-irradiated sample, (b) irradiated by 3 pulses at  $5.5 \text{ J/cm}^2$ , (c) irradiated by 3 pulses at  $1.4 \text{ J/cm}^2$ , (d) after 10 pulses at  $1.4 \text{ J/cm}^2$

## 2.3. Hydrogen permeability of TiN coatings

A bimetallic sample mentioned above, which consists of a rather thick Pd substrate and a thin TiN film, is a convenient system in order to measure hydrogen permeability characteristics of a thin coating. Applicable experimental method was described in details in earlier papers [11-12]. It should, however, be noted that probability of the hydrogen molecule penetration through a thin film to the palladium membrane, during in a single collision process, is in the most cases much lower than that for bare Pd. Hence, a

hydrogen permeability of the bimetallic system can be considered as the hydrogen permeability in the film only, with accuracy up to 10%. Within a frame of this work, there was studied kinetics of the hydrogen penetration through TiN films at temperatures of 630 – 870 K, and at the hydrogen pressure of  $10^{-4}$  Torr. After 10 hrs baking at 870 K, under pressure of  $\sim 10^{-8}$  Torr there was applied the constant volume method. In Fig.3 there is shown a temperature dependence of probability of the hydrogen penetration through a bare Pd substrate (curve 1), and through Pd coated with a 3- $\mu$ m-thick TiN layer (curve 3). For a comparison, there are also shown data for three SS-coated Pd samples (curve 2). It can be seen that a hydrogen permeability of the TiN-coating is one order of magnitude lower than that for an uncoated Pd-membrane, and it is several times lower than that of Pd coated with 3- $\mu$ m-thick SS.

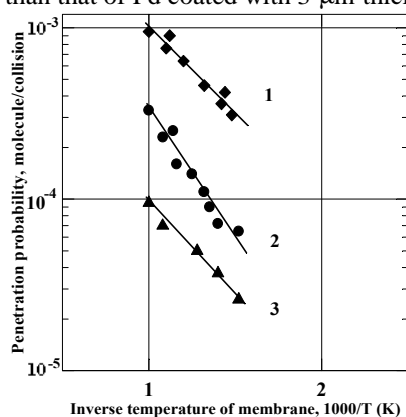


Fig. 3. Temperature dependence of the probability of hydrogen permeation through membrane: 1-bare palladium; 2-3 $\mu$ m SS-coated Pd; 3-3 $\mu$ mTiN-coated Pd

The activation energies of permeability, as one can determine from a slope of the curves presented in Fig.3, were calculated to be 15.5 kJ/mole for bare Pd, 15 kJ/mole for the TiN coating, and 19.9 kJ/mole for the SS-coating, respectively. Measurements of the TiN permeability versus a pressure showed the strong deviation from the  $j \sim p^{0.5}$  law up to  $j \sim p/(1+p^{0.5})$ , where  $j$  represents a rate of the hydrogen permeation through the membrane.

#### 2.4. Outgassing from TiN-coated stainless steel

A block scheme of the experimental facility, as used for outgassing behavior investigations by means of thermal desorption and mass-spectrometry methods, was similar to that described in Ref. [13]. It comprised an SS vacuum-chamber, which contained samples, a monopole mass-spectrometer, and gauges for pressure measurements. The vacuum chamber was connected with a turbomolecular pump and a mechanical fore-pump. The specimens under studies were SS pipes of 32 mm in diameter, 1 mm in thickness, and 140 mm in length. The SS pipes of the identical sizes were coated with TiN-layers. The SS strips of the 150 mm x 10 mm holder construction allowed the samples to be heated up to 673 K, and it provided the sample exposure to a GD hydrogen plasma (of about 200 eV, and  $10^{18}$  ions/cm<sup>2</sup>). Before measurements of the outgassing rate, the 3 hrs baking of the facility at a temperature of 373 K was

performed, and the final pressure  $p_0 = 6 \times 10^{-8}$  Torr was obtained after the system cooling down to a room temperature. After that the investigated sample was heated up to a required temperature value, and the maximum increase in the total pressure, which was caused by gases desorbed from the sample, was measured. At the same time, a mass-spectrum of the gases was registered during the whole thermal desorption process. The specific net outgassing rate ( $q$ ) was calculated from an equation  $q = (p - p_0) S/F$ , where  $S = 50$  l/s was the pumping speed, and  $F$  was an area of the heated sample surface facing the vacuum chamber volume. The measurements were performed with the same system after its 3-hrs baking at 373 K (curves with index a), after a 1-hr hydrogen inflow up to pressure of about  $10^{-4}$  Torr (index b), and after the GD plasma treatment (index c). Fig.4 shows that in a temperature range of 323-523 K the outgassing from the TiN-coated SS pipes and from the U-3M samples were considerable higher than that from the SS samples. The registered mass spectra (Fig.5) showed that the main desorbed gases were those with the ratio  $M/e = 18$  ( $H_2O$ ), 28 ( $CO$ ,  $N_2$ ), and 44 ( $CO_2$ ), respectively. It should, however, be noted that the desorption kinetics changed dramatically after an exposure of the samples to a molecular hydrogen atmosphere under pressure of about  $10^{-4}$  Torr (i.e., a typical  $H_2$  pressure in the U-3M torsatron during the discharge cleaning), or to a hydrogen-plasma of an

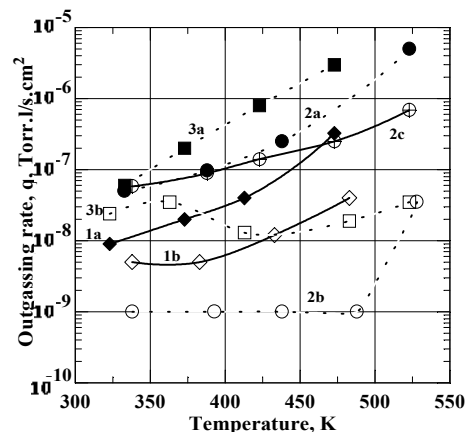


Fig. 4. Temperature dependence of specific net outgassing rate  $q$  for: (1) - stainless steel tube, (2) - TiN-coated stainless steel tube, (3) - TiN-coated U-3M shield sample abnormal GD. In the first case the  $q$  value for the TiN-coated SS showed no change at heating temperatures up to 473 K, and it was considerably lower than that for the SS samples (Fig.4). But, with a further increase in temperature a strong increase in  $q$  value was discovered, up to the values observed for SS without any coating. In that case the main desorbed gases had the ratio  $M/e = 28$ , and 18. It must be noted that, in a comparison with the bare SS samples, a negligible amount of hydrogen was desorbed from the TiN-coated SS samples (Fig. 5). In the second case a considerable increase in  $q$  value was observed after the exposure of the TiN-coated SS samples to GD hydrogen plasma (see Fig.4, curve 2c). In Fig.4 and Fig.6 it can also be seen that a similar character of the outgassing behavior (with some minor differences) was observed for the TiN-coated samples made from the U-3M shield.

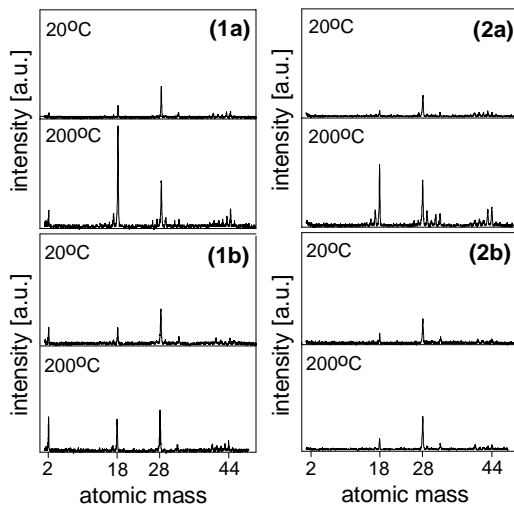


Fig. 5. Mass spectra during baking of stainless steel (1) and TiN-coated stainless steel (2) at 20°C and 200°C after 3 hours heating at 100°C (a) and after 1 hour exposure in  $H_2$  atmosphere at pressure  $10^{-4}$  Torr (b)

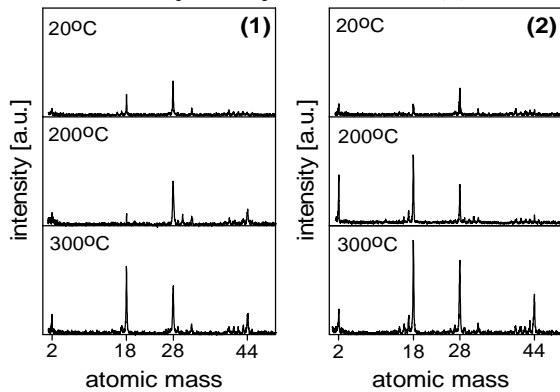


Fig. 6. Mass spectra under baking of TiN-coated samples from U-3M (1) and TiN-coated SS tube after treatment in glow discharge plasma (2)

### 3. Discussion

#### 3.1 Erosion behavior

Erosion behavior and damages of a TiN-coated SS shield under the hydrogen plasma irradiation are determined by various processes, e.g. the arcing, the physical and chemical sputtering, the radiation enhanced sublimation, etc. In general this behavior depends on an energy flux density, a sample temperature, a dose, the impurity concentration, etc. At low flux densities and low sample temperatures (in lack of the effective cooling both parameters are interdependent) the main erosion processes are the arcing and the physical sputtering. The low values of arc ignition probability, which were estimated above, might be due to a low level of the secondary electron emission from the TiN coating. Such a reason had at least been called the main one to explain a considerable increase in the breakdown resistance of a TiN-coated klystron window [5]. Another important reason can also be suggested, e.g. well known influence of the surface cleanness on the arcing probability [14]. After the cleaning by a plasma discharge, probability of the arc ignition upon a boron carbide-surface had been decreased from value of 1.0 (observed for a dirty surface) to less than 0.1 (achieved for a cleaned surface) [8]. Taking into account the results of the biased TiN-limiter experiments, and the obtained TiN outgassing

characteristics, one can suppose that the TiN low outgassing rate could be one of the main reasons of the low arcing level. In order to get a direct evidence of such a mechanism, some additional investigations are needed.

An enrichment of the Ti population in the nearest-surface bulk layer and small change in the TiN-film color (from golden to orange and red only), are main damages observed at a low energy flux density and low temperatures. The first effect can be induced by selective character of the sputtering process, and the second one can depend on the formation of new materials caused by interactions of impurities with the bare coating. When energy flux densities and sample temperatures are increased, a depth of the defective near-surface layer is also increased, and intensive color changes (up to dark-blue color) are observed. Since the U-3M torsatron is an unbaked facility, the main impurity during the plasma discharge cleaning and work plasma discharges constitutes a water vapor. Therefore, the formation of oxides is possible under an intense hydrogen plasma irradiation. Strong oxygen and carbon lines, which were observed during AES investigations of the irradiated TiN samples described in Ref. [2], seem to confirm such a mechanism of the film color changes.

At a high-power plasma flux density some new processes can appear, e.g. the flaking (shelling), the melting, the evaporation etc. A thermal shock can lead to the shelling and flake formation in regions with a lower film-substrate adhesion (Fig. 2c). Some pieces of a film material can be carried over from these parts, and this so-called "shelling" can be the main reason of the high erosion values, as observed for the TiN-coated SS samples during several first plasma pulses. It can also be a reason of a weak dependence of erosion values on the energy flux density (Fig. 1a). At high-energy fluxes (about  $5.5 \text{ J/cm}^2$ ) the TiN film is melted (Fig. 2b) and the film material vapors, and its droplets could give a considerable contribution to the erosion value increase. That was not demonstrated in Fig. 1a, and a reason of that phenomenon could be a screening effect of the target material vapor, similar to that described in paper [15]. Taking into account the TiN film surface density ( $1238 \text{ } \mu\text{g/cm}^2$  [2]) and the particle flux density (about  $10^{17}$  ions/ $\text{cm}^2$  per  $1 \mu\text{s}$  pulse), one can obtain the approximate value of the TiN-erosion coefficient equal to about 10 atoms/ion. This value is considerably higher than that (about 1 atom/ion) measured for bare SS under the similar experimental conditions.

#### 3.2. Permeability

A low value of hydrogen permeability activation energy for the TiN film (15 kJ/mole) and a strong deviation from the known  $j \sim p^{0.5}$  law, usual one for a metallic system, indicate an important role of surface stages in the permeability process (adsorption and dissociation). It also suggests a considerable influence of film porosity on penetration kinetics, as it was observed for some metal coatings deposited upon palladium, by means of the arc sputtering of various cathodes [11-12]. A low hydrogen permeability of the TiN-coating makes this material a perspective one to be

used not only for a reduction of the arcing process upon surfaces of some plasma-device components. Such a coating can also be applied in order to reduce the hydrogen isotopes trapping and inventory by walls of a plasma machine vacuum-chamber. Also a TiN-Pd bimetallic diffusion system, placed inside a plasma device, should be of interest for research on behavior of hydrogen isotopes, e.g. the so-called "tokamakium" - new materials formed upon the internal walls of tokamaks [16] (in torsatrons they could be called "torsatronium"). On the other hand, such membranes could be used for an active control of a hydrogen isotope density near surfaces exposed to high-energy fluxes, e.g. in order to perform some kind of the gas puffing for the surface protection. A potential barrier on the TiN-film and Pd-substrate boundary might be easily overcome, because hydrogen in Pd material is in atomized or partially ionized state, similar to plasma [17]. So, even films with the high-energy hydrogen coupling are not resistible for the hydrogen penetration in such systems.

### 3.3. Outgassing

The measured outgassing rate value for a TiN-coated SS surface has been found to be several times higher than that for the bare SS surface within a range from a room temperature to 500 K (Fig.4). This outgassing behavior differs considerably from the results described in Ref. [4], where the TiN outgassing was estimated to be considerably lower than that for SS in the whole temperature range. A reason for this difference could be a different film composition and structure, caused by different deposition methods and experimental conditions. The observed effect of the insignificant outgassing after a hydrogen inflow (Fig.4, curve 2b), as described in this paper, might have important consequences for a base ultimate pressure value, the particle balance, the hydrogen isotopes recycling and inventory processes in plasma devices, using large TiN coated surfaces. For example, the ultimate pressure in the U-3M torsatron  $p_0 = 4 \times 10^{-7}$  Torr could be reduced several times down to below  $10^{-7}$  Torr by the TiN coating of 100 m<sup>2</sup> area of the vacuum chamber wall surface. As a result of that, one might reduce the out-pumping speed, accordingly. This problem and the results of the outgassing behavior experiments in situ U-3M conditions will be discussed in another paper. This paper considers only one question, arising when large-surface TiN-coated components are used in plasma devices. As it has been shown above, the hydrogen plasma interaction with TiN films can induce drastic changes in their outgassing behavior. The net outgassing rate increases, and a considerable hydrogen amount is desorbed from the TiN coating in the case considered. This outgassing behavior, under/after the plasma irradiation, might lead to changes in plasma performances during experiments.

To explain the considerable outgassing increase after a GD plasma treatment one can suggest the following mechanism. Under a bombardment by neutral atoms and ions, impurities can be rapidly desorbed into plasma and after that they can be partially ionized and partially are pumped out, together with an inflowing hydrogen gas. During this process the TiN surface can be selectively

sputtered, and the Ti enrichment can take place in the coating nearest surface layer. Titanium can actively absorb impurities, and at certain temperatures the formation of oxides, carbides, and another compounds, can occur. If a sputtering coefficient is low (this is usually in order to  $10^{-2}$  per ion under physical sputtering [18]), impurities and new composition materials can be accumulated within the coating surface layer. It can lead to drastic changes in the TiN outgassing behavior, and it can cause damages as well as the film color changes described above.

### 4. Conclusions

The results of the biased TiN-limiter experiments as well as the simulation experiments, which were performed with glow-discharges and plasma accelerators, confirmed the high arc erosion resistance of TiN-coated stainless-steel surfaces. One can combine this effect with the measured low level of the TiN hydrogen permeability and the observed effect of a negligible outgassing from TiN under certain conditions. Hence, one can suppose a wider use of TiN-coated components for the wall conditioning in plasma devices, for an active control of the erosion and recycling processes, and for other technologies. Direct plasma interactions with the TiN-modified surfaces must, however, be excluded in order to eliminate drastic changes in the outgassing behavior and in the coating surface-layer composition.

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