Some problems of plasma-material interactions in fusion devices

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Abstract The paper concerns some questions connected with the interaction of high-temperature plasma-ion fluxes with solid materials inside experimental chambers used for fusion-oriented research, and particularly inside the Torsatron-type facilities. Mechanisms of the boron carbide erosion are described and discussed. Also described is the behaviour of TiN-coated stainlesssteel surfaces under the irradiation with hydrogen plasma-ion fluxes. In summary, there are described damages in the boron carbide layers and changes in the composition of the TiN-coatings.

Key words boron-carbide erosion • plasma-material interaction • TiN-coated stainless-steel

Introduction

Investigations of plasma-material interaction processes are of importance for nuclear fusion physics and technology. The main directions of these investigations are studies of different materials during and after plasma irradiation, as well as research on the behaviour of hydrogen isotopes in plasma facing components and within the whole experimental chamber. As a result of these investigations, various nuclear fusion technologies are now widely used to suppress the plasma interaction with plasma facing materials and to decrease the amount of impurities in plasma itself. This is very important for the hydrogen-isotopes recycling process and for the plasma density control.

To reach some progress in this field within the Uragan-3M (U-3M) and Uragan-2M (U-2M) Torsatron-type facilities, a special programme of plasma-material interaction studies has been realized for several years in the IPP in Kharkov, Ukraine. It includes the use of different modification methods in the manufacturing of plasma facing components, e.g. the application of special TiN coatings and the so-called solid target boronization (STB) processes. There are also applied biased movable limiters and various experimental methods for the simulation experiments and for research on the behaviour of constructional materials. Some studies in this field are performed within the frame of the Polish-Ukrainian scientific cooperation programme, as carried out by researchers from the IPP and those from the Department of Plasma Physics & Technology (P-V) in the IPJ at Swierk, Poland.

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The main purpose of the studies in question [1–6] is to reach a physical understanding of the disruption, erosion, deposition, and transfer processes, occurring on limiter surfaces facing the high-temperature plasma (during biased- and unbiased-experiments). It is needed in order: 1° – to determine the possibility for improvement of the plasma containment, 2° – to investigate the physical properties of various materials, e.g. stainless-steel parts with the TiN coatings and various parts made of $B₄C$ (by its hot-pressing in a vacuum chamber). Some interesting results obtained during recent years have been reviewed and summarized in this report.

Mechanisms of the boron carbide erosion and transfer

Initially, limiters were used only to suppress plasma-wall interactions and to decrease the amount of impurities in the confined plasma. The development of nuclear fusion technology has extended the functional role of the limiters, e.g. the pumped limiters can be used for a plasma density control and the biased limiters can be applied for the removal of the main impurities. Therefore, it was of interest to construct the biased movable B_4C -limiters for the U-3M and U-2M facilities, and to investigate their application for the STB process and for an improvement of vacuum-plasma conditions. To perform such experiments, a movable limiter was first designed and installed in the U-3M vacuum chamber. In the first experiment [1] the limiter consisted of two head-plates of dimensions 90×90×8 mm3, which were placed vertically, as marked with the dashed lines in Fig. 1.

The technology and equipment for the manufacturing of the $B₄C$ limiters were developed in the NSC KhIPT. The $B₄C$ bulk-tiles were made by the hot-pressing of a boron-carbide powder under vacuum conditions. Basic parameters of the $B₄C$ limiters were as follows: the specific density equal to 2.46 g/cm³, the heat conductivity ≈30 W/m/K, and the electrical resistivity $\approx 10^{-2}$ Ωm. An erosion rate has been measured after the irradiation of the $B₄C$ samples with pulsed hydrogen-plasma fluxes. The irradiation was performed

Fig. 1. Schematic cross-section of a torsatron experimental chamber.

within the PROSVET device in Kharkov (during 3 µs expositions) and within the SOWA-400 plasma-accelerator at Swierk (during 1 µs expositions). The measurements gave the following values: the erosion rate ranging from 0.08 to 10 particles/ion, the energy flux varied from 5 to 30 J/cm² shot and the ion energy varied from 200 eV to 2 keV. A specific rate of the out-gassing from the $B₄C$ samples, as measured in a special test after 24 hrs heating at 200°C in a good vacuum (at 4×10^{-9} torr), was $\lt 10^{-11}$ torr l/s cm². That value was considerably lower than the values measured for pure or TiNcoated stainless-steel, those amounted to 10^{-10} torr l/s cm² and 5×10^{-10} torr l/s cm², respectively.

The B_4C limiter was installed within the U-3M vacuum chamber in such a way that its edge could be located at a distance varying from 22 to 12 cm from the plasma column axis. Some measures were provided to apply negative or positive bias-pulses with an amplitude up to 200 V and duration of 1–50 ms. There were performed measurements of currentvoltage characteristics, intensity of the boron BI line and a residual gas composition within the U-3M vacuum chamber by means of a mass-spectrometer. Plasma discharges were characterized by the parameters as follows: the electron density $n_e \approx 2 \times 10^{12}$ cm⁻³, the maximum magnetic field B ≈ 0.46 T, an electron temperature $T_e \approx 10-15$ eV (at the plasma column edge), the plasma pulse duration $t = 50$ ms, and the total discharge power $W \approx 1$ MW. It was observed that intensity of the boron BI spectral line (due to the B_iC sputtering) increases with an increase in the ion current. When a positive bias was applied to the limiter – the boron signal decreased. Therefore such a regime could be recommended for the protection of the limiter against the erosion and for the reduction of impurities amount during plasma experiments. On the contrary, the regime using a negative bias could be recommended for the STB technology. It was not clear, however, which is a mechanism of the boron carbide erosion/deposition and its penetration through a plasma column. It would also be desirable to carry out the boronization procedure during cleaning discharges instead of that during operational plasma pulses.

For these purposes a new version of the movable limiter was installed in the U-3M facility [2]. The main difference was in the horizontal arrangement of the head B_4C -plate (shown by the solid line in Fig. 1). There were two channels for optical measurements of the BI (or OII) and BII spectral lines and eleven collecting probes placed on the plasma facing surfaces of stainless-steel housings of the helical windings. The typical plasma parameters near the plasma column axis during the pulsed discharge-cleaning (PDC) process were $n_e \approx 2 \times 10^{12}$ cm⁻³ and $T_e \approx 10$ -15 eV. The other values were as follows: the magnetic field B ≈ 0.035 T, the pulse duration $= 50$ ms, the pulse frequency $f = 0.2$ Hz, and the total power $W \approx 80$ kW (from 5.4 MHz RF generators). The limiter was biased negatively or positively, up to 200 V, during the biasing pulses lasting 10–50 ms.

With the negative bias the arc discharge was developed with currents ranging up to 2–10 A and there were observed distinct BII-line signals from the plasma volume.

Simultaneously, the OII-line intensity decreased due to the effective trapping of O_2 and H_2O , and the main plasma characteristics showed no changes. It should be noted that the effect was not observed when the positive bias of the limiter (up to 200 V) was applied. It should be noted that arc ignition probability was large (close to 1) during the initial stage of the cleaning procedure (due to a dirty limiter surface). It was considerably lower at the end of the PDC campaign (because of the surface cleaning) even at the maximum negative bias (-200 V) applied to the limiter.

The B_4C arcing erosion was the main reason of a decrease in the oxygen population, as a result of the STB. A new boroncarbide layer, partially coating the protective housings, could effectively absorb water and oxygen molecules. It could prevent an inflow of oxygen from oxides on plasma facing surfaces. Some amounts of positive oxygen ions could also be trapped within a boron carbide tile. It should be noted, however, that it required a few days of the PDC processing to produce a coating of several monolayers of boron on the plasma facing surfaces of the U-3M protective housing. As a result, one could achieve the essential shortening of a discharge cleaning time during the U-3M cleaning campaign.

After about 2×10^4 cleaning discharges, the B₄C-limiter was removed from the plasma edge region and after finishing the experimental campaign the head boron carbide plate was dismounted for detailed investigations. Many craters and spallings of different sizes were observed on that part of the sample surface, which had the direct contact with plasma [3]. Damages of a similar kind, but with lower depth and surface density, were also observed on the sides and even on the back surface of the plate. The most damaged plate edges became those, which during the experiment were located at the smallest distance from the plasma column axis. There were observed not only craters but also partially melted spallings. In contrary, on the sample surface placed far away from the plasma column axis only the craters were found. The majority of those craters had sizes ranging from 0.1 to about 0.5 mm.

Fig. 2. The erosion value per pulse (Q) as a function of the number of plasma pulses (shots).

The main reason of a crater disruption of the boron carbide surface and the near-surface material layers (during the STB) was the arcing process, which was similar to the surface erosion caused by unipolar electrical arcs. There were, however, observed some essential differences. In comparison with the typical unipolar arcs, the first difference was a long duration time of arcing (1–50 ms). The second difference was the immobility of arcs, whose behaviour was unlike that of the unipolar arcs on the metal surface. The effects described resulted in relatively large sizes of the craters, in spite of a rather low temperature ($\approx 50^{\circ}$ C) of the plate surface before the arcing.

Although it was found that the arcs induce a very strong erosion of the boron carbide plate, forming craters and spallings, it was not known what is a physical mechanism of the macro disruption in the form of spallings. Metallographic studies of cuts, which were made perpendicular to the surface of the hot-pressed boron-carbide plate, showed the appearance of two kinds of cracks in the near-surface bulk material within the craters region. The craters of the first kind were "closed", and those of the second type were "opened to surface". The craters of both types were the results of the local overheating caused by arcs.

Taking into account the relatively low thermal conductivity of the boron carbide, the local overheating can lead to a thermal shock and, as a result, it can give a dramatic rise in the thermal stress, leading to the cracking and spalling. Hence, one can deduce that the sparks are the main sources of spallings. It is possible that the spalling of solid-phase boron carbide takes place during the initial stage of arcing. It can be said that in this case the local arcs operate like miniature chisels. It must be noted that a similar spalling mechanism of the hot-pressed boron-carbide disruption was also observed in the simulation experiments, as performed with high-power hydrogen-plasma fluxes produced within the SOWA-400 plasma accelerator. Those experiments showed that the erosion processes are determined not only by interactions between plasma and the bare B_4C -surface, but also by plas-

Fig. 3. The thickness of the shelling layer (d) as a function of the number of the applied plasma pulses.

ma interactions with a "shelling" layer and flakes, which cover the target surface after a few initial plasma pulses. Absolute values of the mass erosion and the thickness of the "shelling" layer in such a case do not depend on the exposure dose, as shown in Figs. 2 and 3. It can be seen that the nature of erosion behaviour practically does not change when the irradiation dose increases by more than one order of magnitude. The points marked by arrows were obtained by calculation after the mechanical peeling of the "shelling" layer and flakes. It is necessary to note that such a (shelling) character of the erosion, caused by the spalling of near-surface bulk material, was not observed for tungsten or graphite irradiated under the same experimental conditions.

Many spalls of different sizes and small droplets of the melted boron carbide, which are shot out from regions of the cathode spots, can penetrate through a plasma column. One could imagine that a boron carbide surface operates like a pellet injector within the plasma device. The ejected particles have relatively high velocities (\approx 100 m/s) and can penetrate far away from the limiter surface. In fact, during numerous experiments there were observed (through optical windows) bright objects moving away from the limiter body. Besides that, intensity of the neutral boron-line emission, as measured within the by BI-line channel, was observed sometimes at a large distance from the limiter. One could suspect that boron vapours from the droplets or melted spalls, which could achieve such a distance, might be the reason of that radiation. It should be noted that the accidental character of an increase in weight of the collecting probes says also in favour of the described mechanism of the boron penetration through the plasma column.

Behaviour of TiN-coated stainless-steel under hydrogen plasma irradiation

The TiN-coatings are often used in the manufacturing of RF-antennas, shields and unmovable limiters for the U-3M and U-2M torsatron facilities. Using such coatings, an essential reduction of the unipolar arc erosion of the antenna surfaces has been achieved. Recently, the experiments performed with a biased TiN-limiter have given the direct evidence of a very low probability of the arc ignition from the TiN-films, on the contrary to the situation observed for the boron carbide layers. The reason of this effect can be the low value of the secondary electron emission from the TiN-layer.

Due to the application of the surface modification technology, which was based on the TiN-coating, controlled plasma discharges were performed using the RF-heating in a quasisteady regime, with the pulse duration up to 50 ms (compare to 3–4 ms with no coating). However, some uncontrollable disruptions of the TiN-coatings on the antenna shields were observed as a result of hydrogen-plasma interactions with the TiN-layer. To protect the first wall, the stainless-steel shields with large TiN-coated surfaces have to be installed within the U-2M machine to be put into operation in the near future. For that reason it was important to explain a mechanism of the disruptions described above. Therefore, it

Fig. 4. The erosion value (Q) *vs.* the distance from the plasma source and *vs.* the number of the performed plasma pulses.

was of interest to study the damaged samples of TiN-coated antenna shields and to investigate behaviour of the TiN-layers in some simulation experiments with hydrogen-plasma pulses.

After the completion of the U-3M experimental campaign, the damaged antenna shield was dismounted for detailed investigations. There were observed some changes in the coating colour from golden (of non-irradiated regions of the shield) to silvery, orange, red or dark-blue. Using the AES, XRD, and fluorescence diagnostic methods [5], it was shown that reasons of the observed colour changes were the various changes in the stoichiometric composition of the TiN surface-layer, and the strong erosion. Preliminary results of research on TiN-coating behaviour, as obtained in the simulation experiments with a plasma treatment by a glow discharge in $H₂$, He, and $N₂$, have shown that the similar colour changes take place. Moreover, it was shown that it is possible to reproduce the primary film colour by an appropriate plasma treatment. The simulation experiments were carried out also using pulsed plasma streams emitted from the SOWA-400 facility at Swierk. It was shown that the erosion of TiNlayer weakly depends on the energy flux density (as shown in Fig. 4), in spite of the different character of disruption processes at higher and lower energy densities.

The TiN-coating erosion values, as measured in those experiments, were a few times higher than those obtained for pure stainless-steel samples. An optical analysis of the surface morphology has shown that the melting of TiN-film occurs at an energy flux density of about 5.5 J/cm2. In the case of the lower energy density (about 1.4 J/cm²), the film melting was not observed. Instead of that, some flakes and small regions of a nude stainless-steel substrate (due to its flaking) were observed. With an increase in the energy flux there was observed a change in the film colour from orange to blackblue one, due to the variations in the surface layer composition. At high energy densities the colour changes mentioned above have not been observed. Maybe, this was due to a decrease in the gas trapping within the film layer, which might be caused by a higher erosion rate. To understand a physical mechanism of such a behaviour some additional investigations are necessary.

Summary and conclusions

The most important results described in this paper can be summarized as follows:

– A new kind of damages of the hot-pressed boron carbide were observed in the form of craters and spallings, when the B_4C samples were exposed to plasma-surface interactions during the discharge cleaning procedure within the U-3M torsatron. It was shown that the cracks at the near-surface bulk of the boron carbide in the region of craters, which were caused by the arcing during the STB processes, are the main reasons of spallings of the boron carbide target. Some spalls or droplets, shot out from the regions of cathode spots of the local arcs, can travel large distances from the solid target. This phenomenon can be used to produce a boron-carbide vapour for the boron-carbide deposition on metallic surfaces placed far from the target, similar to the solid pellet injection. As a result the essential shortening of discharge cleaning time in the Uragan-3M torsatron has been achieved.

– A considerable decrease in the unipolar arc erosion was obtained after the TiN modification of the stainless-steel components facing the plasma (e.g. the RF antennas and shields) within the U-3M torsatron. In the simulation experiments performed with plasma-TiN interactions, and in the biased TiN-coated limiter experiment performed with the U-3M machine, it was shown that the arcing from the TiN-surface is rather low in comparison with that from the stainless steel or boron carbide. The reason of this effect can be a low value of the secondary electron emission from the TiN layer. By means of an optical microscopy, AES, XRD, and fluorescence diagnostic methods, there was also shown that the TiN disruption under the hydrogen-plasma irradiation is accompanied by changes in the stoichiometric composition of the TiN-coating and by its strong erosion. The TiN-film disruption character depends not only on the energy flux **Acknowledgments** The studies described in this paper were partially supported by the Polish State Committee for Scientific Research and the corresponding Ukrainian authorities, since they were performed within the frame of the scientific cooperation agreement between Poland and Ukraine.

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