Stand for coating deposition and coating/materials testing

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Abstract. The paper describes a new laboratory stand constructed for film deposition and for testing of deposited films and materials under pulsed and continuous heat load, ion and electron irradiation. The films are formed on substrates by atoms of target materials as a result of their sputtering by ions of argon plasma. The ion energy and ion flux can be varied independently. This enables the deposition of coatings with variable composition over thickness or of multi-layer coatings. Testing of materials is carried out in plasma under ion or electron irradiation by biasing the tested sample negatively or positively, respectively. The energies of ions or electrons can be varied up to 25 keV. The applied power can reach 4000 W (40 MW/m² power density in the case of a 1-cm² sample) in both continuous and pulsed regimes. In pulsed regime, pulses of 1 – 99% duty cycle at 0 – 500 Hz can be applied to the sample. The pulsed particle load can be combined with a continuous load. The size of the tested sample must not exceed 100 mm in diameter. The heat flux can irradiate the whole sample or be focused at its center (minimum spot of ~ 4mm²). Heating of the samples up to 2800 K is possible. At the same time, the backside of the tested sample could be actively cooled. This paper presents the results of deposition and testing of a B₄C coating on tungsten and tungsten testing.

1. Introduction
Coatings with different functional properties are widely used in different branches of technology. They could provide corrosion resistance, could be used as protective coating, could work as diffusion barriers, etc. In particular, a protective coating could be necessary for tungsten tiles of the ITER divertor. A number of studies [1,2] showed that tungsten under high-power density heat and particle loads undergoes various effects, including cracking, erosion, macro-scale particle emission, melting-point lowering. These and other effects could lead to a rapid destruction of the divertor tiles and generation of tungsten dust. The B₄C coating was proposed as a protecting in-situ renewable coating for tungsten tiles of the ITER divertor [3]. Until now, methods of B₄C coating deposition in plasma have not been investigated.

Material testing under intensive plasma and particle irradiation is being performed on a number of special devices and facilities. Quasi-stationary plasma accelerators, such as QSPA-T [4] or QSPA Kh-50 [5], provide sample irradiation by pulsed plasma fluxes with a power density of up to 5 GW/m² and a duration of less than 1 ms. The time-gap between the pulses is about several minutes, and this fact

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leads to testing series of about a hundred cycles. A high heat-load can be obtained in devices with an electron beam, like JUDITH II [6]. In this machine, an electron beam with a power of up to 200 kW is used, but it scans the surface of the sample being tested and produces an average value of the heating load. The devices with ion beams extracted from a high-current discharge, like Magnum-PSI [7], MARION [8] and PISCES [9] can provide a power density of up to 20 MW/m² with a duration of up to hundreds of seconds.

Combining stationary and pulse heat loads is a challenging task and is achieved only in the Magnum-PSI device by periodically ramping up the discharge current to higher values. At present, stationary irradiation is possible only for 100 sec in this device, while in ITER a 1000 sec discharge is expected. All the devices mentioned are of high complexity and require complex maintenance by skilled personnel.

A special device is needed for the development of a coating deposition technique (in particular, B₄C coating deposition on ITER divertor tiles) and for testing of coatings with high particle and heat loads. The paper presents a device, which meets both of these purposes.

2. Experimental device

The stand allows deposition of coatings by means of sputtering of targets by ions of an argon gas discharge with a heated cathode. The advantage of this type of discharge is the possibility of ion irradiation in a wide range of ion energies (from tens of eV to tens of keV) and by various ion fluxes. Therefore, it becomes feasible to try a large amount of various regimes of deposition and choose the best one. By replacing the sputtered target with the tested sample, one can irradiate the latter by ions or electrons of the plasma.

The Stand for COating Deposition and MATerial Testing (CODMATT) is presented schematically in figure 1 ((a)-coating configuration, (b)-testing configuration). The stand has a vacuum chamber (1) with a cylindrical plasma chamber (2) inside it. The plasma discharge is ignited between a heated tungsten cathode (3) and an anode (4). The anode is designed as two concentrically ring-shaped plates with a ring-shaped slit between them. Ions are drawn off from the discharge area through the slit of the anode. The bigger anode plate can be moved by the motion feedthrough (5). The discharge current can reach 20 A with the voltage between anode and cathode being 200 V. The residual pressure is 10⁻⁶ Pa. The main residual gas components are H₂O (75%) and H₂ (25%), the other components being O₂, N₂ and CO₂ (<< 1%). A quadrupole mass-spectrometer is used to measure the composition of the residual and working gasses. The vacuum chamber is equipped by two lock chambers. The upper lock chamber (6) is used for the substrate manipulator; the bottom lock chamber (7) is used for target installation and the tested sample manipulator. The lock chambers are separated from the main vacuum chamber by gate valves (8). The manipulators move through the sealings (9). The stand control is automated.
2.1. Coating configuration

The coating configuration is presented schematically in figure 1 (a). The substrate for coating (10) with a size of $15 \times 15 \times 1 \text{ mm}^3$ is mounted on a manipulator (11) equipped with a motorized drive (not shown). The motorized drive provides an automatic and simple substrate exchange procedure and transport of the substrate into the vacuum chamber. The manipulator is water-cooled, which allows fixing the substrate temperature in the range of $300 \text{–} 900 \text{ K}$ during the deposition process. The temperature is measured by a pyrometer. Before and during deposition, the substrate can be irradiated by plasma ions for achieving better adhesion and better structural properties of the film, respectively. The main working gas is argon because it provides a high sputtering rate. For investigation of hydrogen (or some other gas) trapping during coating, the mixture of argon and hydrogen (or other investigated gas) can be used as a working gas.

The target (12) is irradiated by ions extracted from the plasma. Two anti-dynatron electrodes (outer – 13 and inner – 14) are located concentrically around the target. The anti-dynatron electrodes suppress the ion-electron emission from the target. These electrodes are also needed for ion beam formation. The anti-dynatron electrodes and the target are biased by a voltage of $\leq 25 \text{ kV}$ by means of the electric feedthroughs (15). The maximum power supplied to the target is 2500 W, to the inner electrode, 500 W, and to the outer one, 1000 W. The target and the electrodes are mounted on the water-cooled target manipulator (16).

The coating is formed on the substrate by atoms sputtered from the target by plasma ions. For better adhesion, creation of an interlayer between the substrate and the coating is needed. It is possible to form this layer with a composition different from that of the target by sputtering the anti-dynatron electrodes. This can be achieved by varying the shape of the ion beam (by moving the electrodes and the anode), as well as by varying the value of the electrode potential. In the case of depositing $\text{B}_4\text{C}$ onto tungsten, the outer electrode is made of tungsten, the inner one is made of graphite, and the target is a mixture of boron and graphite. The deposition procedure is as follows. First, substrate sputtering is conducted, then the tungsten electrode is sputtered (tungsten atoms are being deposited onto the substrate), after that, the graphite electrode is gradually biased (W-C layers are deposited) and, lastly, sputtering of the target begins, while the sputtering of the electrodes is minimized (boron carbide is deposited).

Due to the automatic control of power supply, one may handle the ion fluxes by a predetermined program and carry out the deposition in an automatic regime, including preliminary ion cleaning of the substrate, deposition of the sublayer and of the coating. The deposition rate of $\text{B}_4\text{C}$ was measured as $\approx 10 \mu\text{m}/\text{h}$.

2.2. Testing configuration

The testing configuration is presented schematically in figure 1 (b). Using the stand, material testing under high heat and particle loads in stationary and pulsed regimes can be conducted. In the testing configuration of the stand, both the target and the anti-dynatron electrodes are replaced by the sample (17) on the sample holder (18). The testing sample manipulator (19) is actively cooled. By using different systems of sample holders, the temperature of the sample during testing can be varied in the range $600 \text{–} 2800 \text{ K}$. In thermal cycling tests, ions or electrons irradiate the backside of the tested sample.

The ion irradiation provides a maximum energy release in the surface layer of the sample. Moreover, the possibility exists to reveal the influence of ion irradiation and implantation on the behavior of materials and coatings under thermal cycling. The electron irradiation leads to a widening of the surface area of energy release. At the same time, in this case surface sputtering is excluded and radiation-induced defects formation in the area of energy release is reduced sharply.

During the testing of materials, the sample holder is biased by a voltage of $\leq 25 \text{ kV}$ (power of $\leq 4 \text{ kW}$). The irradiation power can be varied during the testing process. The pulse duration of cycling irradiation exceeds 1 ms, while the duty cycle assume any value in the range $1 \text{–} 100 \%$. The diameter of the tested sample is limited to 100 mm. By altering the anode and the sample positions, various ion...
beam shapes can be obtained and, hence, various surface areas can be irradiated (from 4 mm$^2$). This regime can also be used for modification of the materials surface by ion or electron irradiation.

**3. Results**

As part of a pilot program of the CODMATT testing, experiments with boron carbide deposition on tungsten substrates were carried out. Also, thermocycling tests of the deposited coatings were carried out in order to verify their adhesive properties. To test the coatings of B$_4$C in the T-10 tokamak, it was required that samples with coating thickness of a few microns withstand thermal cycling up to 900 K.

An analysis of the concentrations of the components in the coating and control of the integrity of the coatings after thermal cycling was carried out by the electron microscope TESCAN VEGA 3, which had a console to perform elemental analysis by X-ray energy-dispersive spectroscopy (EDS). Targets formed with 4:1 boron-graphite powder mixture were used for deposition of the coating. The substrate temperature during deposition did not exceed 600 K. The B/C concentration in the coatings deposited was found to be in the range 3.8 – 4.2. The SEM image of a coating after deposition is presented in figure 2 (a). These coatings were tested by thermocycling in the 450 – 1200 K range. After 150 heat cycles, small cracks of a dimension of 0.1 µm with a distance between them around 200 µm appeared. The SEM image of a coating after thermocycling is presented in figure 2 (b). After 100 more cycles, these cracks did not widen and no flake forming was observed. The cross-section of a crack (figure 2 (c)) shows that the crack goes through both the coating and tungsten substrate. One can conclude that the cracking of the coating was caused by substrate cracking in the first place.

![SEM images of a coating before thermocycling](image1)

![SEM images after 150 heat cycles](image2)

![FIB crosssection of the crack](image3)

**Figure 2.** SEM images of a coating before thermocycling (a), after 150 heat cycles in the range 450 – 1200 K (b) and a FIB crosssection of the crack (c).

In the material test mode, tungsten samples were irradiated. For these experiments, ITER divertor tungsten tiles were used as samples with dimensions of 20 × 20 × 1 mm$^3$. The samples were prepared by electro-spark cutting, then polished with abrasive polishing paper having a grain size of 2 – 3 microns and washed with ethanol in an ultrasonic bath.

The samples were irradiated by H$_2^+$ ions ($E = 10$ keV/at and a flux of $1.2 \times 10^{18}$ at/m$^2$s). The irradiated zone was in the shape of a ring with a diameter of 10 mm located in the center of the sample. The pulse duration was 10 s and the irradiated zone had a temperature of 1500 K after each pulse. The samples were not actively cooled in the experiment. After the end of the pulse, the sample was cooled down for 20 s to 900 K. After each series of irradiation, the sample surface was studied by SEM. On the irradiated surface, shallow holes formation with 20 µm dimensions and deep cavities were observed. Their position changed and size increased as the number of cycles of irradiation reached 430 pulses.
The sharp edges of the holes, their considerable depth and their shape indicated that their formation was not the result of ion sputtering. On the same side, constant sample heating at the same temperature with a cumulative time of the pulses did not lead to any significant surface relief modification. Interestingly, formation of similar holes on the surface of fused quartz during irradiation by helium ions was observed in [10]. Often, their appearance was related to the growth of whiskers. The paper concludes that their formation, as well as the occurrence of whiskers, are a consequence of the diffusion fluxes and mobility of dislocations under the conditions of ion irradiation. One may suppose that during tungsten irradiation, the same processes lead to surface modification. Cyclic ion bombardment at high temperatures stimulate the processes leading to their formation in the material at a macroscopic depth and the appearance of holes in the irradiated surface.

4. Conclusions
A simple and convenient to operate automated stand was implemented. The stand allows one to investigate the regularities of formation and deposition of single- and multi-component coatings. Also, the stand allows one to study the behavior of materials under thermocycling and high-density ion and electron irradiation.

During pilot testing of the CODMATT stand, a $B_4C$ coating was deposited on tungsten and pulsed ion irradiation of tungsten samples was performed by 20 keV $H_2^+$ ions. The results of these tests are presented and discussed.

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