

CONFERENCE PRE-PRINT

**ASSESSMENT OF B₄C AS FIRST WALL COATING
FOR THERMONUCLEAR REACTOR**

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Abstract

The new ITER baseline includes tungsten (W) as first wall (FW) material. This increases the risk of increased plasma contamination by W (and other metallic impurities) and the associated plasma radiation that may decrease the achievable Q. Together with the ITER Organization (including activities in the frame of TA IO/24/TA/4500000207) R&D is performed for study B₄C coating that can be applied to first wall panels and other metallic areas in line of sight of the plasma in ITER to reduce influx of high-Z metallic impurities in ITER to prevent this risk from materializing.

1. INTRODUCTION

Boron carbide is a material or component for the manufacture of many industrial products as it has high hardness and wear resistance, high elastic modulus, high chemical stability, low Z, low density, high erosion resistance and high melting point. Presently, boron carbide is of interest as a protective material in the form of a coating on the tungsten first wall of a thermonuclear reactor [1,2]. The fact is that spattered tungsten enters the plasma and reduces the efficiency of its retention and could lead to plasma radiating cooling in a thermonuclear reactor [1,3]. Therefore, there is a need for a protective layer on tungsten to prevent it from entering the plasma. In this regard, the researchers drew attention to B₄C, which also has a low absorption capacity to hydrogen and is resistant to thermal shocks. In this connection, the problem arises of choosing an effective technology for creation of the B₄C layer on tungsten. Here, thermal spray technologies such as plasma spraying, detonation spraying, etc., attract attention as they demonstrate a fairly high productivity. Of these technologies, plasma spraying is probably the most widespread and it has been already used for spraying boron carbide [1,4,5].

Detonation spraying (DS) is one of the most effective gas thermal technologies [6]. Based on the phenomenon of gas detonation [7], this technology enables producing wear resistant, electro-insulating, surface protecting, and other functional coatings on surfaces of parts from different materials. Unlike other thermal spray technologies, in which the spraying process is continuous, DS is a pulsed process, so that spraying is performed by a series of powder shots fired by a detonation gun. The advantage of this process is that by adjusting the time intervals between shots, the heating of the substrate is controlled and overheating can be avoided. DS was chosen in present studies for spraying boron carbide onto tungsten substrates, since it has already been tested in spraying refractory tungsten [8] and pure (without metal-binder) chromium [9] and tungsten [10] carbides. In present work, detonation spraying was performed on the CCDS2000 Detonation Spraying Facility developed at the Lavrentyev Institute of Hydrodynamics Siberian Branch of RAS.

The ITER-like W samples with different dimensions and with applied B₄C coating were used for exposure on:

- the experimental setup of the BETA complex at the Budker Institute of Nuclear Physics (BINP SB RAS). The experimental setup used for simulation of thermal loads is based on the industrial laser IPG YLS-4000U, detailed facility description and heat fluxes parameters are provided in [11];
- the quasi-stationary high-current plasma accelerator QSPA-B (JSC SRC RF TRINITI). The facility generates axial pulsed flows of deuterium plasma and allows to reproduce plasma thermal loads expected during transient processes (ELM-like) in ITER. Similar QSPA-T facility description and heat fluxes parameters are described in [12].

The paper includes description of experimental results obtained on mentioned above facilities and conclusion considering further usage of B₄C coating (deposited by detonation spraying) at tokamaks.

2. DEPOSITION OF B₄C COATINGS BY DETONATION SPRAYING – TECHNIQUE DESCRIPTION

The spraying unit of the Detonation Gun (DG) is a barrel with a powder feeder and a gas distributor mounted on a vertical stand. Under computer control, during spraying, the barrel can move up and down on the stand, and the manipulator rotates and/or moves horizontally the processed part. The design of the device ensures the safety of work and effective control of spraying parameters. Fig. 1 shows the principle of DG operation.

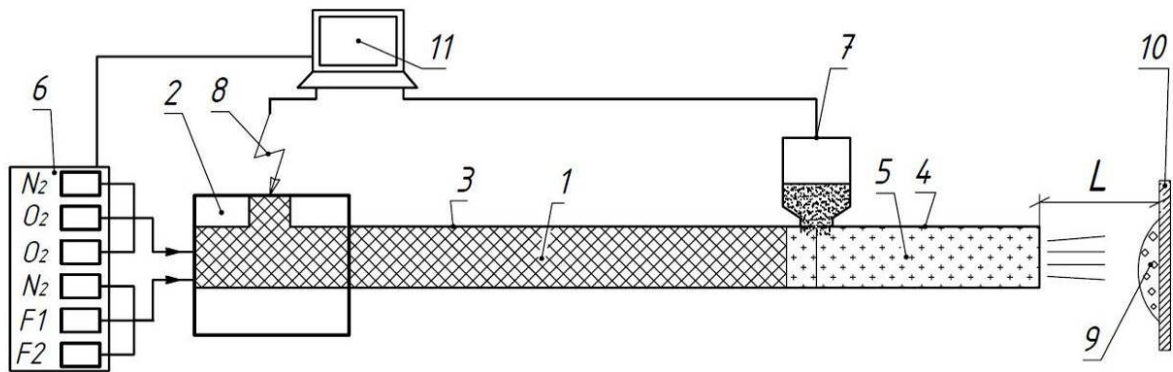


FIG. 1. Detonation spraying layout for CCDS DG: 1 – fuel + oxidizer detonating gas mixture, 2 – mixing-ignition chamber, 3 – breech section of the barrel, 4 – muzzle section of the barrel, 5 – nitrogen (purge gas), 6 – gas distributor, 7 – pulse powder feeder, 8 – spark plug, 9 – coating, 10 – substrate (processed part), 11 – control computer. F1, F2 – fuel supply (2 ports), O₂ – oxygen supply (2 ports), N₂ – nitrogen supply (2 ports), L – spraying distance.

As noted above, detonation spraying is a pulsed process consisting of a series of cycles (shots), in each of them, the barrel of the detonation complex is filled with a gas explosive mixture, a portion of powder is injected into the barrel, detonation of the explosive mixture is initiated and after firing the barrel is purged with nitrogen or air. The detonation products of the explosive mixture accelerate powder particles up to 1000 m/sec and heat them up to the melting state. After leaving the barrel, particles collide with the processed part and form a coating.

The task was to produce samples with coatings of 50 and 1000 microns thick on tungsten substrates (tiles) of various areas and thicknesses according to Table 1. These samples should be subjected to further studies on the possibility of using B₄C coatings on the first wall of a thermonuclear reactor.

TABLE 1. LIST OF TUNGSTEN SUBSTRATES FOR SPRAYING

№	Sprayed surface area, mm ²	Substrate thickness, mm	Coating thickness, μm	Comments
1	7 x 7	2	50 ± 5	Will be used in experiments for further imitation conditions of stationary process low energy plasma flux (~ 10 's eV plasma temperature)
2	15 x 15	2	50 ± 5	
3	up to 24 x 24	up to 10	up to 50 ± 5	Were used in experiments for imitation conditions of non-stationary process
4	up to 24 x 24	up to 10	1000 ± 50	

The process of producing coated samples includes preparing the surface of the substrate, measuring the thickness of the substrate, performing DS and measuring the thickness of the resulting sample in order to control the thickness of the B₄C layer. The condition for obtaining a sufficiently strong bond of the coating to the substrate (adhesion) in DS is to create a surface relief of the substrate with a roughness size of the order of the size of the sprayed particles. The most common is sandblasting using special equipment. However, experiments have shown that in result of surface treatment with corundum on a sandblasting facility, a certain amount of embedded Al₂O₃ particles remain in the surface layer of the substrate. The number of embedded particles can be reduced by applying erosive treatment of the tile surface with a jet of particles accelerated in the DG barrel due to more precise control of the particle velocity in the DG CCDS2000. Erosion treatment on DG also has the advantage that this operation and the subsequent spraying operation are performed from a single mounting of the tile on the manipulator, which increases the productivity of the coated tile manufacturing process. Therefore, the method of erosion surface treatment of tiles on DG was chosen for the manufacture of research samples. In addition, in order to exclude the presence of unwanted materials in the coating-substrate interface, erosion treatment was carried out using boron carbide powder with a particle size of 50-80 microns. The measurements of the thickness of tungsten tiles before and after coating deposition were performed using the digital micrometer GRIFF (Cyber-Instrument LLC, Moscow, Russia) with a measurement accuracy of 1 micron.

Boron carbide powder GOST 5744-85 with an approximate grain size of M28 according to GOST 3647, the average particle size of about 20 microns, was used for spraying. According to the powder certificate, it contains 96.54 wt. % B₄C and impurities in the form of free boron (0.14%), free carbon (1.70%), B₂O₃ (0.16%) and Fe (0.16%). In result of validation experiments, the technological parameters of spraying were defined.

Manufactured samples were subjected to destructive testing, Fig. 2 shows optical images of the coating microstructure obtained using an OLYMPUS GX-51 metallographic microscope (Olympus Corporation, Japan).

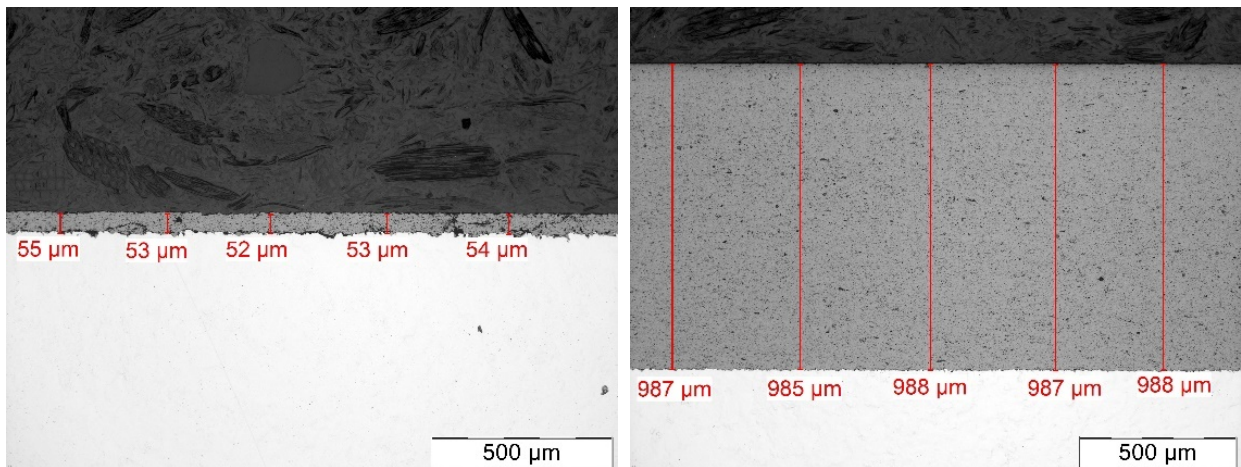


FIG. 2. Coatings with a thickness of 50 ± 5 (left) and 1000 ± 50 (right) microns on a tungsten substrate.

It can be seen that the coating has a fairly dense homogeneous structure. Previous studies have shown that B₄C coatings produced by DS had a porosity < 20%, a hardness of 13.9 ± 1.6 GPa, and a cohesion of 12.4 ± 0.6 MPa. In this case the porosity of the coatings was determined from the analysis of the cross-sectional optical images using OLYMPUS Stream Image Analysis software Stream Essentials 1.9.1 (Japan).

2. ITER TRANSIENT THERMAL LOADS SIMULATION (LASER EXPERIMENTS)

The experimental setup for simulating transient thermal loads, based on the industrial IPG YLS-4000U laser, is equipped with a two-color pyrometer, which allows monitoring the dynamics of the sample surface temperature. A schematic diagram showing the arrangement of the main components of the setup, examples of the spatial distribution and temporal dynamics of the heat flux W_s is presented on Fig. 3. Examples of the spatial distribution and temporal dynamics of the heat flux W_s are shown in Fig. 4 and Fig. 5, respectively.

During the experiments, two heat flux values were implemented: $W_s \approx 0.46 \pm 0.07$ GW/m² and $W_s \approx 0.2 \pm 0.03$ GW/m². This load is equivalent to ~50% and ~20% of the peak heat flux of ITER-relevant ELMs or ~5% and ~2% of the peak heat flux of unmitigated disruptions (given a required mitigation factor of 90% [13]). The sample surface was examined using SEM between pulse series; after the experiments, metallography was performed to analyze subsurface damage.

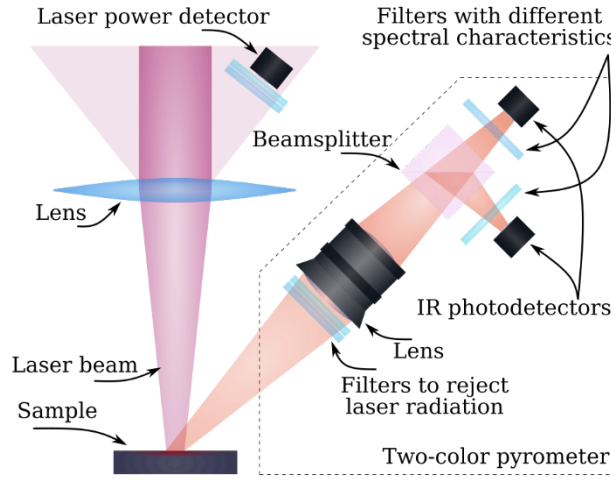


FIG. 3. Schematic of the BINP experimental setup for transient heat load simulation, showing the main components layout.

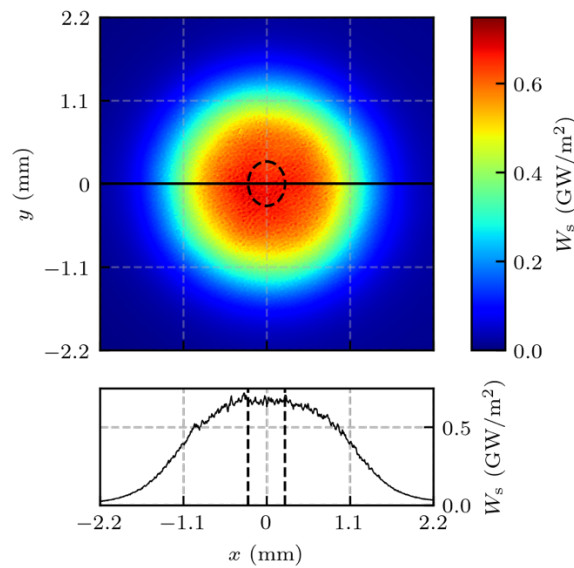


FIG. 4. Spatial distribution of laser heating on the sample surface. The temperature measurement area is outlined with a dashed line.

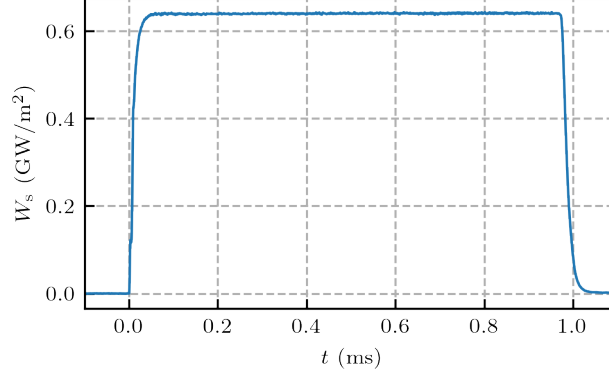


FIG. 5. Dependence of the heat flux W_s on time t during a laser pulse with a duration of $t_h \approx 1$ ms.

Summary of the B₄C coating testing results under transient thermal loads is presented on Fig.6: it is shown the surface damage processes observed during the experiments (cracking, loss of substance, melting, and coating detachment), the heat load (characterized by heat flux W_s and the heat flux factor F_{hf}), and the number of pulses. Up to 25% of the plasma energy can be deposited on the first wall panels by ELMs (for the ITER tokamak, the peak heat flux is $W_s \sim 1$ GW/m² with a duration of $\Delta t \sim 0.1 - 1$ ms [17]) [14-16]. The results of the work carried out at the Budker Institute of Nuclear Physics show that a transient thermal load corresponding to $\sim 50\%$ and $\sim 20\%$ of the ITER-relevant ELMs peak heat flux leads to significant erosion of the boron carbide coating (up to detachment). Another process causing substantial heating of plasma-facing components is a plasma disruption (for the ITER tokamak, the peak heat flux is $W_s \sim 10$ GW/m² with a duration of $\Delta t \sim 2 - 5$ ms [17]). The results indicate that even with the required ITER disruption mitigation factor of 90% for the heat load [13], such events will lead to significant coating erosion.

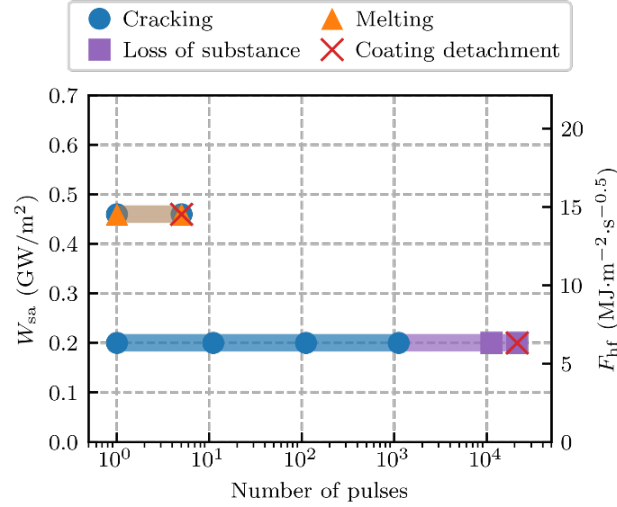


FIG. 6. B₄C coating damage mapping under transient thermal loads.

3. ITER TRANSIENT THERMAL LOADS SIMULATION (PLASMA STREAM EXPERIMENTS)

The testing of the coated samples was carried out at the quasi-stationary high-current plasma accelerator QSPA-B. The facility generates axial pulsed flows of deuterium plasma and allows to reproduce plasma thermal loads expected during transient processes in ITER, a schematic diagram of sample irradiation at the facility is shown in Fig.7.

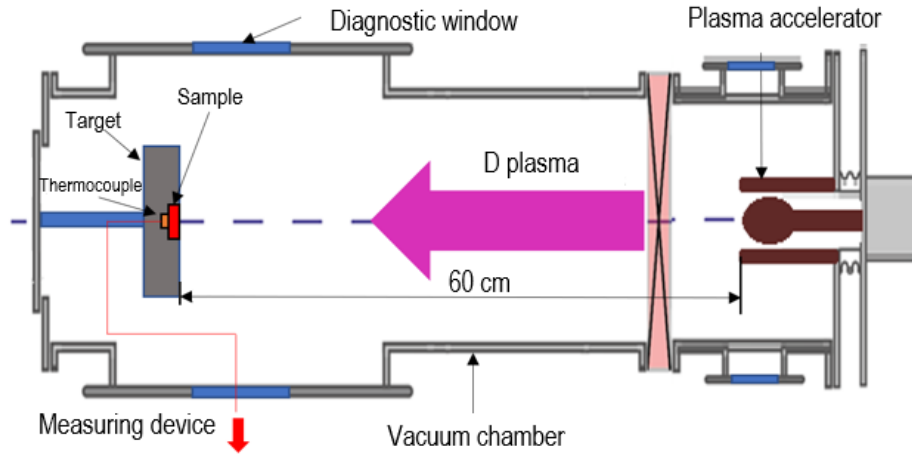


FIG. 7. Irradiation scheme at the QSPA-B

The samples were irradiated in two modes: for samples with a coating thickness of 100 and 250 μm , the heat load (Q) was 1.0 MJ/m^2 , and for 1000 μm , it was 1.3 MJ/m^2 . The exposure duration in experiments was 1 ms. The number of pulses (N) varied from 1 to 50. An example of the spatial distribution of the heat load across the surface of a flat target is shown in Fig. 8. It was obtained using a thermocouple calorimeter, the sensitive element of which was a molybdenum target plate.

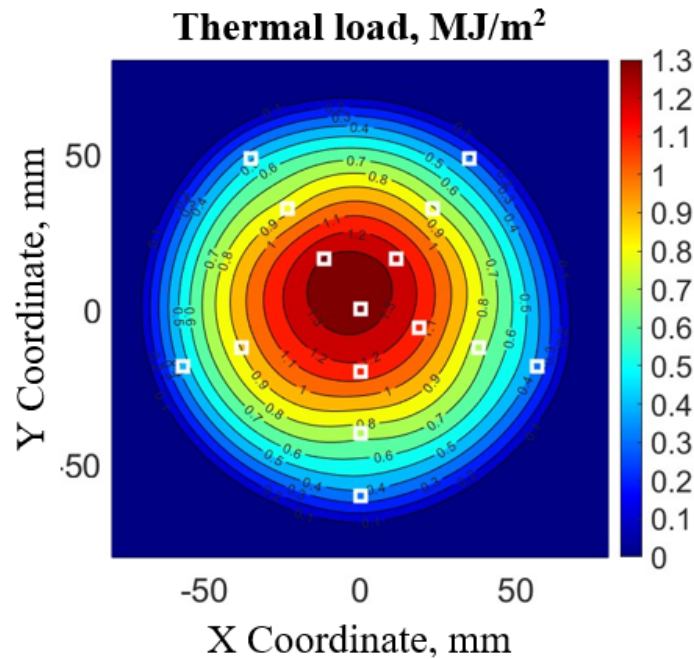


FIG. 8. Spatial distribution of thermal load during irradiation of the target sample

The paper is mainly focused on obtained results for erosion rate of a boron carbide coating at the QSPA-B facility. The mass loss was determined by weighing the samples before and after exposure. Based on this data, the average amount of coating thickness change per pulse was calculated at boron carbide's porosity of 18% (density 2 g/cm^3). The results are presented in Table 2, Table 3 and Figure 9. With a thermal load of 1.3 MJ/m^2 and a coating thickness of 1000 μm , the average erosion rate was 11 $\mu\text{m}/\text{pulse}$. At thermal loads of 1 MJ/m^2 , and coating thicknesses of 100 and 250 μm , the maximum erosion rate occurred immediately after the start of the experiment, then decreased to 5 $\mu\text{m}/\text{pulse}$.

Important note is that the erosion rate 5 $\mu\text{m}/\text{pulse}$ (after first pulses) is the same for samples with different B₄C coating thickness.

TABLE 2. MASS LOSS AND THICKNESS CHANGE PER PULSE FOR TWO SAMPLES WITH A COATING THICKNESS OF 1000 μm .

$h, \mu\text{m}$	S	Q	Δm 0-5	Δm 5-10	Δh 0-5	Δh 5-10
1-1000	5.6	1.3	8.62	10.34	7.7	9.2
2-1000	5	1.3	15.12	11.68	15.0	11.6

TABLE 3. MASS LOSS AND THICKNESS CHANGE PER PULSE FOR TWO SAMPLES WITH A COATING THICKNESSES OF 100 AND 250 μm .

$h, \mu\text{m}$	S	Q	Δm 0-1	Δm 1-3	Δm 3-10	Δm 10-30	Δm 30-50	Δh 0-1	Δh 1-3	Δh 3-10	Δh 10-30	Δh 30-50
100	4.9	1	66.40	4.45	-	-	-	67.3	4.5	-	-	-
250	5.5	1	56.00	5.25	5.54	6.60	4.80	50.6	4.7	5.0	6.0	4.3

$h, \mu\text{m}$ – boron carbide coating thickness, $Q, \text{MJ/m}^2$ – thermal load; S, cm^2 - surface area; $\Delta m, \text{mg/pulse}$ – change in the sample's mass; $\Delta h, \mu\text{m/pulse}$ – changing the coating thickness B_4C at porosity of 18% (at a density of 2 g/cm^3).

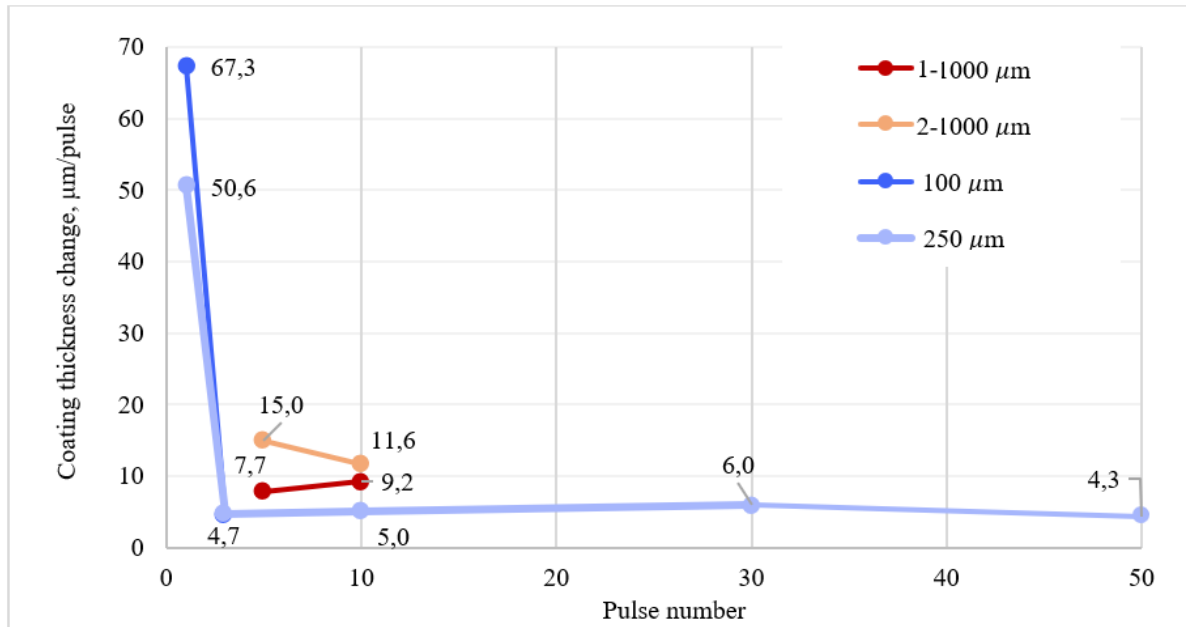


FIG. 9. The rate of erosion of boron carbide coatings with thicknesses of 100, 250, and 1000 μm when exposed to plasma fluxes at the QSPA-B facility (at porosity of 18% and density of 2 g/cm^3).

CONCLUSIONS

During the research, optimal modes of detonation spraying of B_4C coatings on tungsten substrates (tiles) of various sizes were developed. Spraying was performed on the CCDS2000 Computer Controlled Detonation Spraying Device (CCDS2000 Detonation Gun). The performed studies allow us to assert that the technology of detonation spraying is a fairly productive technique for practical application in the manufacture of protective coatings from B_4C .

First experiments performed at facilities for transient thermal load simulations have shown that B_4C coating produced by detonation spraying will not survive during up to 100 shots at significant heat loads that's why practical application of such ceramic coatings in fusion devices will require not only the development of effective ELM and disruption mitigation systems but also technologies for *in situ* repair of damaged coating areas. Additional High Heat Flux testing at electron beam gun facility are also planned.

Further experiments with low energy plasma flux (~ 10 's eV plasma temperature) for imitation conditions of stationary process will provide important information about sputtering rate of the coating, gas trapping and changes in composition and morphology of the coating surface during irradiation – these results could have critical

impact on decision whether B₄C coating produced by detonation spraying is suitable as coating for plasma facing components at tokamak application.

Work is performed partly in the frame of TA IO/24/TA/4500000207 with ITER Organization.

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