

Evaluation of vacuum plasma-sprayed boron carbide protection for the stainless steel first wall of WENDELSTEIN 7-X

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Abstract

To minimize radiation losses of the plasma during long pulse operation, the first wall protection of WENDELSTEIN 7-X demands low-Z plasma facing materials. In addition to carbon materials on high heat flux loaded components, 300–500 µm thick vacuum plasma-sprayed (VPS) layers of boron carbide (B₄C) are considered as coatings on large actively cooled stainless steel panels. In order to evaluate the behaviour relevant to the expected plasma wall interactions, an extensive material characterization and test programme was executed. Also included is the examination of the industrial manufacturing and the suitability of these coatings as 70 m² first wall plasma facing material for W7-X. To improve the adhesion of thick B₄C layers on stainless steel, different interlayers have been investigated. Based on the results, a VPS-based coating technique was identified which is suitable to manufacture the B₄C protection layers on stainless steel wall panels of W7-X.

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1. Introduction

Protective coatings for first wall plasma facing components (PFC) made of stainless steel have been developed for the W7-X stellarator, which is presently under construction. In contrast to the high heat flux loaded divertor target plates, the design of the approximately 120 m² wall protection strongly depends on the shape of the plasma vessel and the minimum plasma-wall dis-

tance and the resultant heat load [1]. The heat load (thermal radiation and neutrals at plasma operation with high densities) on 70 m² of the outboard wall protection is reduced to 100–200 kW/m². Together with a relatively low curvature of the vessel, larger cooling structures can be provided [2]. Laser welded, embossed panels made of austenitic stainless steel (SS) and covered with VPS-B₄C layers as low Z material offer efficient and reliable manufacturing, installation and operation. Based on various encouraging results concerning the characterization and application of B₄C in fusion experiments, e.g. [3–5], an extensive development programme was started to investigate the plasma-interactive and thermo-mechanical behaviour of industrially

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Fig. 1. Prototype of a full scale mock-up made by BUCO (Germany) and VPS-B₄C covered by PLANSEE (Austria). The double wall panel made of stainless steel is Laser welded and embossed.

manufactured B₄C covered SS panels for the particular operating conditions in W7-X. The preferred VPS-B₄C coating with an SS interlayer on a full scale mock-up (Fig. 1) and the following heat flux tests completed the development program. This article summarizes the results of the plasma-physical and thermo-mechanical investigations.

2. Vacuum plasma spraying process of B₄C coatings on stainless steel

The major advantage of the plasma spraying technology is the high velocity obtainable (up to 500 m/s) and the high temperature achieved in the plasma jet (up to 15000 K). This makes it possible to melt even the most refractory material. The melted powder jet forms a coating consisting of many layers of overlapping thin lenticular particles. VPS is used for materials which are sensitive to oxygen such as metals, carbides, nitrides and borides. For application of B₄C coatings, the VPS technique provides layers with low contents of impurities and residual gas, especially the content of oxides and O₂ is reduced.

The strong mismatch in the coefficient of thermal expansion (CTE) between stainless steel substrates and B₄C coatings limits the achievable thickness of coatings [6]. This makes it necessary to apply intermediate layers to reduce the residual stress, in order to produce a pure VPS-B₄C layer of 300–500 μm without delaminations. The interlayer should compensate the differences in thermal expansion. This can be accomplished by selecting a material with a thermal expansion coefficient between those of substrate and coating. In the case of the VPS-B₄C coating, molybdenum and alternatively a mixture of B₄C and SS were chosen as bonding layers. The development was focused on SS interlayers to avoid

high Z impurities in the plasma in case of damaged PFC. Furthermore the stainless steel substrate has been actively cooled during the spraying process in order to keep the substrate at low temperature to guarantee a low stress situation after coating.

3. Characterization of VPS-B₄C layers

3.1. Material properties

A programme to evaluate the plasma-physical and thermo-mechanical properties of B₄C on water-cooled stainless steel (1.4301) panels was carried out. The measured material properties of VPS-B₄C that are of interest for the described application are summarized in Table 1.

Surface impurities on the coatings were identified by EDX. Additional measurements by ion beam analysis (RBS) confirmed the EDX results. The main impurities found by EDX on the present VPS coatings were oxygen and iron. The reduction of the amount of impurities, compared to earlier samples of VPS layers [6], is a result of the development process.

Microindentation tests of VPS-B₄C coated samples were performed with Vickers indenter on metallographically polished cross-sections. Young's modulus, Universal and Vickers hardness of the VPS sprayed B₄C

Table 1
Selected material properties of VPS-B₄C

Property	Value, unit	Remarks
Composition		
B	78*–85 at.%	EDX, *RBS
C	15–20* at.%	
Impurities		
O	1.0–2.0* at.%	
Fe, Ni	<0.2 at.%	
Melting point	2720 K	[7]
Density	2270 (2520) kg/m ³	[6]
Porosity	5–35 %	Image analyse
Surface roughness R_{max}	30–80 μm	Profilometry
Young's modulus	220–270 GPa (290–450)	
Vickers hardness HV	2900–3200 N/ mm ²	
Thermal conductivity	(27–29) W/m K	
20 °C	2.0 W/m K	[6]
500 °C	2.8 W/m K	
Specific heat capacity		
20 °C	980 J/kg K	
500 °C	1865 J/kg K	
Electrical resistivity	~3.5 Ω m	

Brackets mark values of monolithic B₄C [7].

coating were measured. For the determination of the Young's modulus a Poisson's ratio of 0.18 was assumed.

The thermal diffusivity of the VPS-B₄C coating was measured by the laser flash method. With the additionally measured specific heat capacity the thermal conductivity was calculated.

3.2. Physical properties

3.2.1. Sputtering behaviour and deuterium retention

The sputtering yield of the VPS-B₄C coating was measured under bombardment with deuterium ions as a function of the energy of the incident ions at room- and slightly elevated temperatures due to the bombardment (for 1–3 keV up to 100–230 °C). The comparison with values for monolithic B₄C material shows only a small difference in the scatter between these values and the data for the VPS-B₄C coating (Fig. 2).

The trapping of 1 and 8 keV deuterium implanted into polished bulk B₄C has been studied at room temperature up to fluences of 6.7×10^{19} D/cm². The saturation concentration of deuterium in B₄C is 0.42 D/(B+C) and almost identical to D implantation in graphite [10].

3.2.2. Dielectric properties of B₄C coatings at 140 GHz

For the characterization of the microwave properties of B₄C-coated components at the ECRH frequency of W7-X ($f = 140$ GHz), investigations of samples with varying thicknesses ($d = 50$ – 300 μm) on metallic substrates (Cu, Mo, TZM and SS) were carried out. To determine the real and imaginary part of the dielectric constant of the coating, ϵ_1 and ϵ_2 , the reflection coefficients R_p and R_s for polarization parallel and perpendicular to the plane of incidence were measured as a function of the angle of incidence α at 140 GHz, and, for $\alpha = 20^\circ$, as a function of frequency. From a fit to theory [11,12], ϵ_1 and ϵ_2 can be determined unambiguously. Additionally, the reflection coefficient averaged over an angular range of $0 < \alpha < 90^\circ$ and both polarizations was deduced. This coefficient is an essential parameter for the heat load induced by stray radiation from the ECRH heating system [13]. Results for various coatings are summarized in Table 2.

Table 2

Dielectric parameters ϵ_1 , ϵ_2 , reflection coefficient at perpendicular incidence as well as angle-averaged reflection coefficient for various samples

d -B ₄ C (μm)	Substrate	ϵ_1	ϵ_2	$R (\alpha = 0)$	R av.
50	TZM	–	–	1.0	0.92
100	TZM	6.9	20.8	0.54	0.58
150	Cu	5.9	6.0	0.33	0.49
190	TZM	22.5	7.6	0.63	0.60
300	Mo	10.9	16.6	0.47	0.50
300	SS	–	–	0.46	0.46
300	SS	29.0	8.6	0.29	0.38

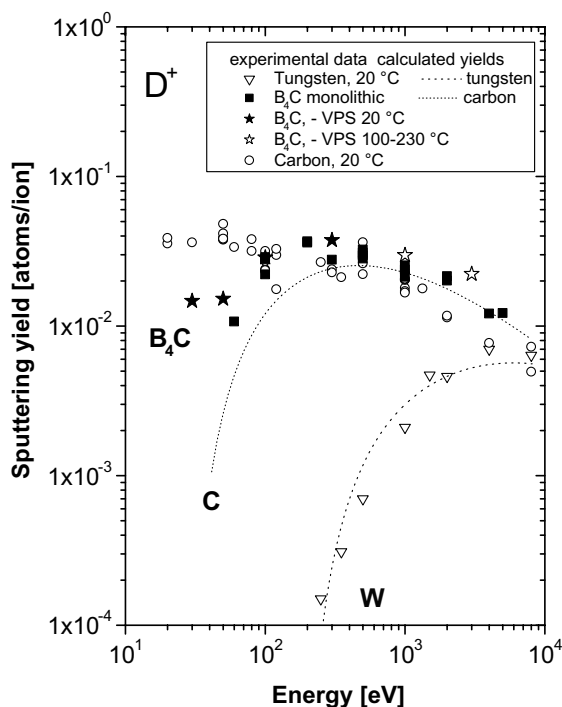


Fig. 2. Comparison of measured sputtering yield of VPS-B₄C coating, monolithic B₄C, carbon and tungsten under bombardment with D. Dashed lines show the calculated yields [8]. Data of monolithic B₄C, C and W [9].

The analysis of the data shows: For thin coatings ($d < 200$ μm), the parameters depend strongly on the thickness of the layer. A reason of the variation of the material properties could be contaminations in the layers, especially metallic ones, as even small contents can strongly alter the dielectric properties.

For the case of incomplete absorption of the ECRH beams in the plasma [13], the low reflection coefficient will lead to high thermal loads of the B₄C-coated panels in W7-X. Especially in the range 150–200 μm, resonant absorption can occur, which leads to an absorption of up to 90%. For this reason, wall protection elements

with high reflectivity (like graphite) will be used close to the ECRH heating ports.

3.2.3. Plasma exposure in fusion devices

To support the investigation of the plasma-physical behaviour of the VPS-B₄C coatings, in the tokamak TEXTOR, 10 poloidal carbon limiter blocks were replaced by inertially cooled Cu blocks, coated with 170 μm B₄C. The blocks were exposed as limiter in direct plasma contact with heat loads up to 8 MW/m². No severe cracking or delaminations of the protection layer were observed [14]. However, the following surface analysis showed large amounts of arc traces, which burned through the coating down to the Cu substrate [15]. This phenomenon cannot be transferred to the expected environment of the W7-X wall protection, where the material will be used near the vessel wall, without direct plasma contact, being loaded by very low fluxes of charged particle and power only. In order to estimate the probability of arcing under first wall conditions, B₄C coated samples were exposed at the vessel walls of the stellarator W7-AS and the tokamak ASDEX Upgrade. The position of the samples was similar to the expected plasma-wall distance in W7-X. The total exposure time in ASDEX Upgrade was ≈2350 s (444 discharges) and 643 s in W7-AS (1445 discharges). The optical and electron microscopic analysis of the ASDEX Upgrade and W7-AS wall sample surfaces showed no visible arc traces, cracks or delaminations.

The operation of B₄C coated ICRH faraday rods in ASDEX Upgrade for more than 10 years without arcing problems supports the above-mentioned observation. The position of the faraday rods is 1 cm behind the protection limiters. In 2001 an electron microscopic analysis of removed rods did not reveal any arc traces. Only erosion of the B₄C layer was found.

In a direct relation to the arcing behaviour of these nearly ceramic protection layers, the influence of B₄C layers on the behaviour of glow discharge cleaning in W7-X was evaluated [16]. Glow discharge experiments were performed. The coating of the panel does not influence the discharge in any visual manner. No problems will be expected for the glow discharge cleaning on first wall protection partly B₄C coated.

4. Examination of B₄C/stainless steel composite

4.1. Evaluation of the thermo-mechanical behaviour

4.1.1. Residual stress measurement of the coating

Operational failure of thermally sprayed layer composites are mainly due to critical operational stresses superimposed by residual stresses and thermal and mechanical fatigue during cyclic load.

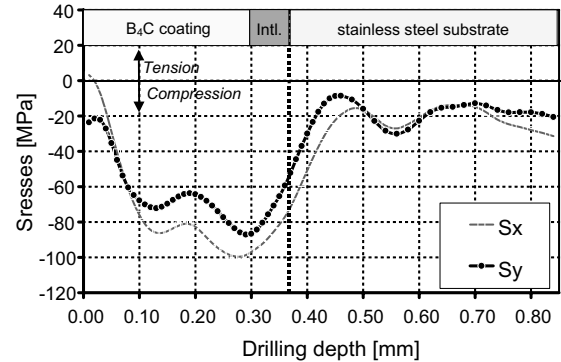


Fig. 3. Residual stresses in the VPS-B₄C coating (~300 μm), molybdenum interlayer (~70 μm) on stainless steel obtained by the microhole drilling method.

By using the microhole drilling method, residual stress fields can be measured over the drilling depth with appropriate resolution. In several drilling operations a circular, cylindrically shaped microhole is brought step by step into the component surface. The residual stresses in the component are locally relieved due to material removal, deform the surface around the drilled microhole and are measured using high resolution strain gauge rosette for every drilling step. Using calibration curves and material properties data (Young's-, shear modulus) the measured surface strains are converted into nominal strains at the bottom of the drilled hole for every drilling step.

The residual stresses in various substrates with VPS-B₄C coatings and interlayers were measured up to a depth of 0.86 mm. Fig. 3 shows an example of a measured stress distribution. An isotropic stress field (x, y) over the drilling depth was determined. Compressive stresses were measured in the coating, with a maximum value of ~100 MPa. In the substrate surface small compressive stresses of about 25 MPa can be found. In Fig. 3, two strong stress gradients can be observed, one in the first 10 μm and the second at the interface. The first one can be explained due to uncertainties of the method at the outer surface, some porosities and structural defects as well as by the surface roughness. The strong stress gradient at the interface of this composite is related to the different thermophysical properties of coatings and substrate.

These measurements give qualitative but not fully quantitative stress results. 80–140 MPa compressive stress in the B₄C coatings on SS substrates can be assumed.

4.1.2. Evaluation of the stress situation in the coating by FEM and strain gauge measurement

For the chosen design of the wall panels the operational stresses and strains have been analysed under

critical operating conditions of W7-X. The subject of interest is the stress situation in the coating and bonding layer. At first, the behaviour of the coated panels with inner hydraulic pressure and simultaneously applied thermal load has been simulated by FEM without including the residual stress situation to get an overview about the stress and strain situation during operating in W7-X. In a second step, the measured residual stress field can be superposed to the FEM results. To verify these computer-simulations, small-scale mock-ups (SSM) have been tested with hydraulic and thermal loads.

For an inner pressure of 15 bar (without thermal load) the FEM calculation of the principal stress on the surface of a coated SSM resulted in a tensile stress of 105 MPa. The calculated stress is in good agreement with strain gauges measurements of the principal stress of about 110 MPa at room temperature. In case of the maximum heat load on the tested SSM (500 kW/m², $p = 5$ bar), the calculated principal stress in the coating increases to ~ 150 MPa. Taking into account the compressive residual stress of the order of 100 MPa, only minor tensile stress in the coating will be expected for the operating of the panels in W7-X.

4.1.3. Thermal tests

Six SSM with different coatings were extensively tested in the First Wall Test Facility (FIWATKA) at FZ Karlsruhe. 1000 cycles of 3 min each at 500 kW/m² (factor 2.5 of the expected maximum heat flux on W7-X panels) were applied on each sample. During the experiments, the highest temperature reached on the B₄C coating surfaces was ~ 300 °C at 500 kW/m² [17].

The final thermal cycling test of a VPS-B₄C coated full scale mock-up (Laser welded and embossed) was performed at 200 kW/m² heat load and 1221 cycles. No damages on the coating or plastical deformations of the element occurred.

4.2. Metallographical examination

In order to guarantee the reliability of the selected coating parameters, all the heat load tested elements were metallographically examined. Especially the inspection of the B₄C coated samples with SS or Mo interlayers showed no cracks or delaminations. The chosen spraying parameters resulted in homogeneous B₄C layers which are completely bonded to the substrate. Separation of layers were not observed. Fig. 4 illustrates these results for the preferred layer combination.

The full scale prototype was cut for the metallographical examination and analysed in the horizontal midline. The results demonstrate the lateral homogeneity of quality and adhesion of coating and interlayer on a full scale wall prototype.

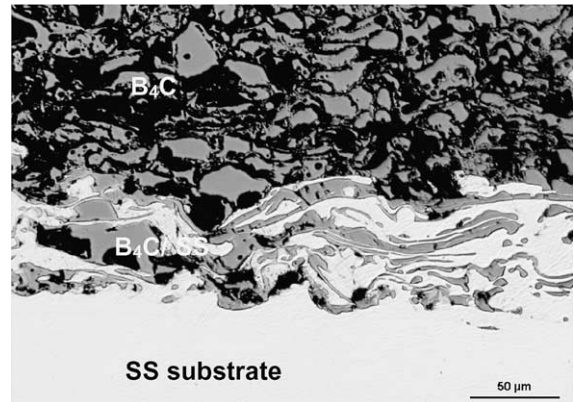


Fig. 4. Micrograph of a VPS-B₄C/SS mixed interlayer on an actively cooled SS substrate figures.

5. Conclusions

The plasma-physical and thermo-mechanical evaluation of industrially manufactured VPS-B₄C coatings on watercooled stainless steel panels demonstrates the suitability of B₄C as low Z first wall plasma facing material of W7-X.

The metallographical examinations and the measured material properties show that a reliable VPS-B₄C coating with mixed SS interlayer can be realized on the W7-X wall panels. This coating system with a thickness of 300–500 μm ensures a reliable adhesion, erosion resistance and acceptable dielectric properties at 140 GHz. The mixed B₄C/SS interlayer avoids high Z impurities in the plasma in case of damaged PFC.

Laser welded and embossed panels made of SS and covered by VPS-B₄C coatings allow an efficient and reliable manufacturing, installation and operation for 70 m² outboard wall protection in W7-X.

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