ELSEVIER

Journal of Nuclear Materials 307–311 (2002) 89–94

journal of nuclear materials

www.elsevier.com/locate/jnucmat

Characterisation and thermal loading of low-*Z* coatings for the first wall of W7-X

D. Valenza [a], H. Greuner [a,*], G. Hofmann [b], S. Kötterl [a], J. Roth [a], H. Bolt [a]

[a] *Max-Planck-Institut für Plasmaphysik, EURATOM-Association, Boltzmannstr. 2, D-85748 Garching, Germany*

[b] *Forschungzentrum Karlsruhe, EURATOM-Association, Institute of Applied Thermo- and Fluid Dynamics, P.O. Box 3640, D-76021 Karlsruhe, Germany*

Abstract

Low-*Z* coatings with a thickness up to 500 μm are being developed as plasma facing material on stainless steel first wall panels for the W7-X stellarator under construction at Greifswald, Germany. The materials under investigation are boron carbide (B_4C) and a silicon–boron–carbide (SIBOR, manufactured from Plansee A.G., Austria), both applied by vacuum plasma spraying. Thermal loading was performed in the First Wall Test Facility (FIWATKA) at the Research Centre Karlsruhe. In particular, stepwise increasing heat loads from 50 to 500 kW/m^2 and cyclic heat loads up to 1000 cycles of 3 min duration were applied to characterize the thermo-mechanical behaviour of the different coatings. Additionally, 2D and 3D finite element modelling is used to support the experiments and to predict the failure threshold of the coatings, which is also verified experimentally.

1. Introduction

For the Wendelstein 7-X stellarator (W7-X), which is under construction in Greifswald, Germany, protective ceramic coatings are being developed and will be applied on the plasma facing first wall panels. The total first wall surface area of W7-X is approximately 120 m^2. The major part of the internal outboard area of the W7-X vacuum vessel will be protected by actively cooled stainless steel panels (Fig. 3) coated with a low *Z* (atomic number) ceramic material (B_4C or Si(C)). The internal inboard area of the vacuum vessel will be protected with graphite tiles clamped on Cu alloy cooling structures. The aim of these low-*Z* coatings is to inhibit interaction of the stainless steel surfaces with the plasma, which would lead to high-*Z* impurity influx into the plasma. Because W7-X will be operated in steady state with discharge durations up to 30 min, impurities could accumulate in the plasma, leading to unfavourable radiation and energy loss of the plasma.

* Corresponding author.
E-mail address: henri.greuner@ipp.mpg.de (H. Greuner).

The plasma spray (PS) technique offers the possibility to coat 3D curved surfaces with materials with very high melting temperatures at reasonable costs. Among the different techniques, 'vacuum plasma spray' (VPS) is the most promising method for applying the coatings on the plasma facing surfaces of the first wall panels. This coating process ensures sufficient coverage of the plasma facing surfaces with the low-*Z* coating for a lifetime throughout the whole operational period of W7-X.

2. Coating materials requirements, coating technology and coating characterisation

The low-*Z* requirement of the coating materials comes from the energy loss of the plasma by radiation, which is proportional to Z^4, with Z being the atomic number of the plasma impurity. Potential candidate materials for first wall coatings, which have already partly been investigated [1], or are under investigation in the present study, are boron carbide (B_4C) and a recently developed, material based on the silicon–boron–carbon system (e.g. SIBOR, powder mixture of 88% Si, 10% B, 2% C [2,3]). SIBOR is manufactured by plasma

0022-3115/02/$ - see front matter © 2002 Elsevier Science B.V. All rights reserved.
PII: S0022-3115(02)00939-X

spraying of a pre-sintered powder, thus combining the advantages of SiC (high bulk thermal conductivity, coefficient of thermal expansion higher than B_4C, therefore better adapted to the stainless steel substrate) with the possibility of plasma spraying through the addition of boron for producing a liquid phase. As plasma facing material for W7-X, however, B_4C is favoured, owing to its lower Z number and the better oxygen gettering of B in comparison to Si. The plasma facing material of the first wall will be subjected to erosion from energetic plasma particles (ions and neutral atoms). The required thickness of the coating is 300 μm for SIBOR and 500 μm for the B_4C coating to allow for an erosion lifetime of the plasma facing material throughout the experimental operation of the device. The substrate material for the W7-X first wall components is stainless steel AISI 304 with a thickness of 5 mm between coating and the confined water coolant. During normal operation in W7-X the surface temperature should not exceed 400 °C. The stationary thermal flux during normal operation is 100 kW/m^2 with local excursions up to 200 kW/m^2 [4], thus the thermal conductivity of a 500 μm thick coating has to exceed 0.5 W/m K. In order to prevent failure by cracking or detachment, the adhesion and cohesion strength of the coating has to exceed 35 MPa (tensile). The surface roughness is limited to $R_a \leqslant 8$ μm, impurities below 500 appm (excluding gases).

The VPS technique has been chosen for the deposition of the coatings because it offers a good compromise between manufacturing costs and quality of the coating (low impurities and residual gas, good thermo-mechanical properties). Adhesion and cohesion of the coating are key parameters for the successful application of thick coatings. PS-B_4C coatings without bonding interlayer have been only deposited up to a thickness of approximately 0.2 mm on large surfaces [5,6]. However, by applying a graded bonding layer between B_4C and steel substrate, thus providing a continuous change in material properties like thermal expansion and compliance, the thickness of the B_4C layer on top of the bonding layer could also exceed 1 mm [7]. The aim of the present work was to develop highly adhesive coatings with and without interlayers in the thickness range up to 0.5 mm. First results of the characterisation of VPS coatings [8] have shown that the VPS technique provides a ceramic coating with low impurity content (<2000 ppm), low porosity (<15%) and thermo-physical properties which satisfy the W7-X requirements (thermal conductivity higher than 2 W/m K at RT) [4].

3. Thermo-mechanical experiments

The thermo-mechanical behaviour of coated components with geometry relevant to the W7-X panels was investigated in the First Wall Test Facility FIWATKA at the Research Centre Karlsruhe [9]. The surface heat fluxes under cyclic thermal loading where chosen according to the normal operation conditions of W7-X. Also experiments were carried out at heat fluxes well above the W7-X values.

The FIWATKA device consists of a large water-cooled vacuum vessel made of stainless steel, with a diameter of 1.8 m and a length of 2.4 m (Fig. 1). Inside the vacuum vessel, a graphite heater with a surface area of 0.6×0.4 m^2 is installed. The heater is surrounded with water-cooled copper shields, which allow the aperture of test windows of adjustable size for the installation of test specimens and mock-ups. The mock-ups can be connected to an independent cooling water circuit. The water temperature and pressure at the mock-up inlet can be adjusted from room temperature to 100 °C, and from 1 to 6 bar. Two sets of mock-ups were prepared for the experiment. The first set consists of eight 'small-scale mock-ups' ('SSM', Fig. 2), which are made of water-cooled stainless steel substrates of the dimensions $300 \times 80 \times 13$ mm^3. Six of the eight SSM were coated (VPS) with 18 different coatings resulting from the combination of materials (B_4C and SIBOR), different thickness (500, 300, 100 μm) and various interlayer systems (without, Mo, coating materials + stainless steel mixture).

Temperature sensors were installed in the mock-ups at three different locations: two points on the cold backside surface and at the end of a hole drilled into the heated steel side, below the coatings (Fig. 2(a)). In two mock-ups strain gauges were also installed on the back surface of the cold side (Fig. 4, scheme). The second set consists of two 'full-scale mock-ups' (FSM, Fig. 3), which are stainless steel (SS DIN 1.4301) prototypes of a W7-X first wall element. The average length of these elements is 550 mm, the width 300 mm and the thickness 14 mm (without coating). The FSM are actively water-

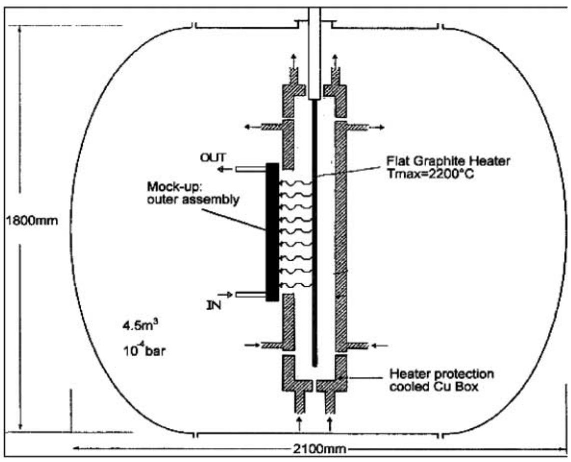

Fig. 1. Scheme of the FIWATKA device.

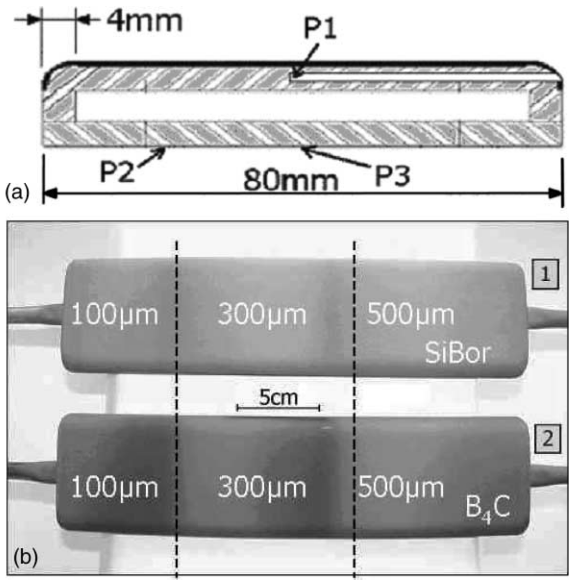

Fig. 2. (a) Small-scale mock-up cross-section, (b) small-scale mock-ups coated with SIBOR (1) and B_4C (2).

cooled and one of the two FSM was coated with B_4C of thickness 200 μm (VPS).

The SSM were tested with steady state heat fluxes ranging from 50 up to 500 kW/m^2. For each load, two cooling water flows were applied, 1 and 2 m/s with inlet temperature 35 °C and pressure 5 bar. The duration of the constant heat load during each step was about 15 min. At the end of the steady state loading, a cycling load (Fig. 3) was applied on the two most promising coatings: B_4C + (SS-B_4C) interlayer and SIBOR + (SS-SIBOR) interlayer. Thousand cycles of 3 min each (in total about 50 h), at 500 kW/m^2 and with 1 m/s water flow, were applied. During the experiments, the highest temperature reached on the B_4C coatings surface was ~ 300 °C at 500 kW/m^2. No plastic deformation was observed during the thermal cycling experiment.

In general, it appears that the coatings with interlayer provide a larger safety margin and allow the application of 500 μm layer thickness due to a reduced residual stress field in comparison to the coatings without interlayer. This statement is also confirmed by other evidence such as observed delamination during the water-jet cutting of the mock-ups, after the experiments. In a few cases the coatings without interlayer detached from the substrate, while the coatings with interlayer did not.

The strains (Fig. 4) and deformations taking place in the mock-up during the experiment were quite small, also due to the intrinsic geometrical stiffness of the samples. The highest measured tensile strain was smaller than 100 μm/m in the stainless steel. From the finite element analysis, the highest tensile strain, taking place in the B_4C, coating is about 1400 μm/m and in the stainless steel, in the position where the strain gauges were applied, about 100 μm/m.

At the end of the SSM test campaign, no failure (i.e. cracking or delamination) caused directly by the heat loads was observed on the coatings with interlayer. Only in the SSM coated with B_4C, in the region of 500 μm coating thickness, a large piece of coating (80 mm long and 3 mm wide) chipped-off along the edge. The origin of this failure seems to be related to the manufacturing process, after which already fine cracks were observed in the region of later delamination.

The FSM were tested with steady state loads ranging from 50 to 500 kW/m^2 in the case of the uncoated mock-up, and from 50 to 200 kW/m^2 in the case of the B_4C coated mock-up. The cooling water inlet temperature was 50 °C (max cooling water outlet temperature 80 °C) and the maximum surface temperature was ~370 °C on the uncoated mock-up. The FSM experiment showed also a good thermo-mechanical behaviour of the B_4C coating. The horizontal displacement of three points ($S1$, $S2$, $S3$, Fig. 5) was measured during the experiments. Point 2 is located at the centre of the mock-up and points 1 and 3, which lie on the same vertical plane containing point 1, are ~190 mm distant from the point 2. A deformation parameter S was defined as $S = S2 - (S1 + S3)/2$. With the highest heat load 500 kW/m^2, the maximum extrapolated deformation of the mock-up edges would have been about 5 mm (i.e. the mock-up

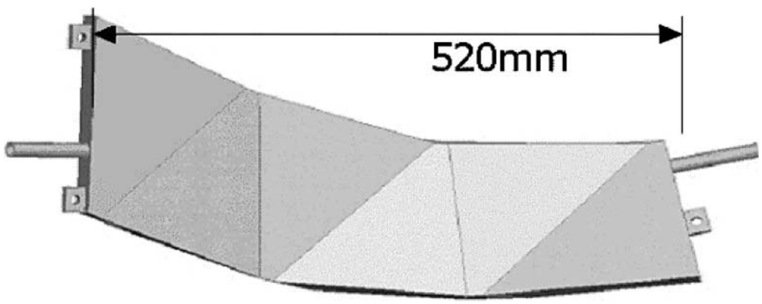

Fig. 3. Full-scale mock-up of a W7-X outboard first wall panel.

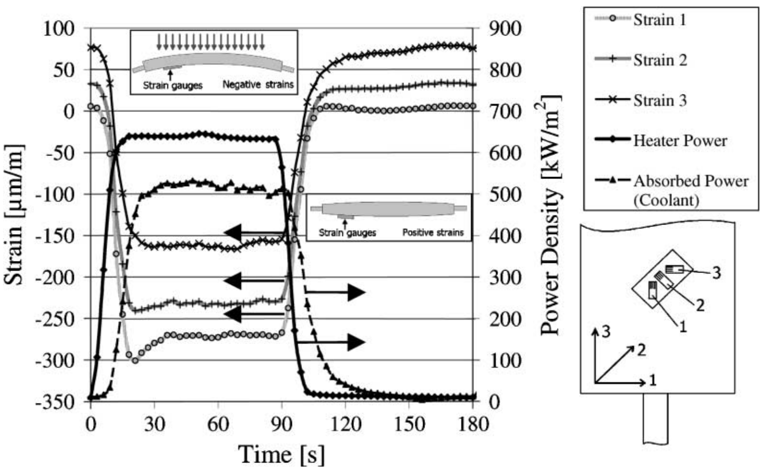

Fig. 4. Example of load cycle of a small-scale mock-up coated with boron carbide. The measured strains and heat fluxes are plotted in function of the time.

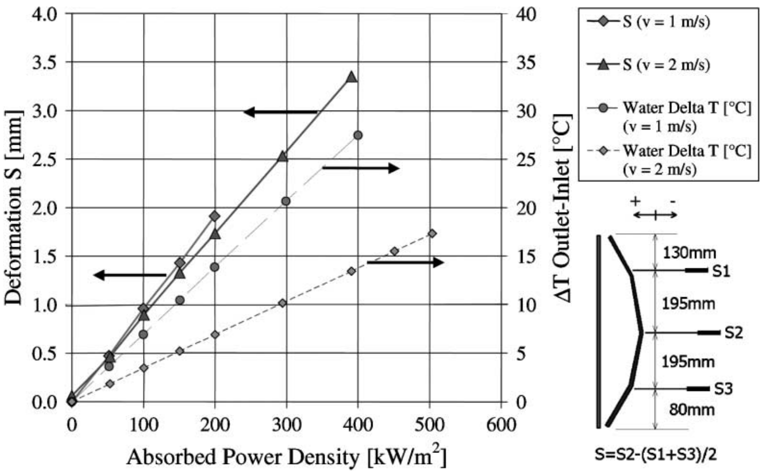

Fig. 5. Deformation of the FSM in function of the absorbed power and cooling water temperature difference between inlet–outlet for different water flows.

elongated) with respect to the central part of the mock-up. To avoid coating failure due to excessive deformation, the heat load in the experiments with the coated mock-up was limited to 250 kW/m^2.

4. Finite element analysis

In support of the FIWATKA experiments finite element analyses have been conducted. Two models, reproducing the SSM with B_4C coating (500 μm thick) and the FSM without coating, were generated and the experimental steady state loading conditions were simulated. In the SSM thermo-mechanical analysis the following boundary conditions were applied to the model: 500 kW/m^2 uniform heat flux, 30 °C inlet water temperature, 5 bar water pressure. In the FSM thermal analysis the boundary conditions were: 500 kW/m^2 average heat flux (not uniform), 50 °C inlet water temperature.

The SSM analysis result shows that coating regions at the sample edges are the most critical because the highest stresses and stress gradients develop at these locations (Fig. 6, highest von Mises stress above 200 MPa assuming a B_4C Young's modulus of 100 GPa [10]). The maximum temperature, also located in those regions, is slightly below 300 °C. In the 500 μm thick B_4C coating the temperature difference between the surface and the interface with the stainless steel is about 70 °C. The calculated stresses of this analysis do not yet take into account the residual stress field that arises in the coating during the deposition process. The maxi-

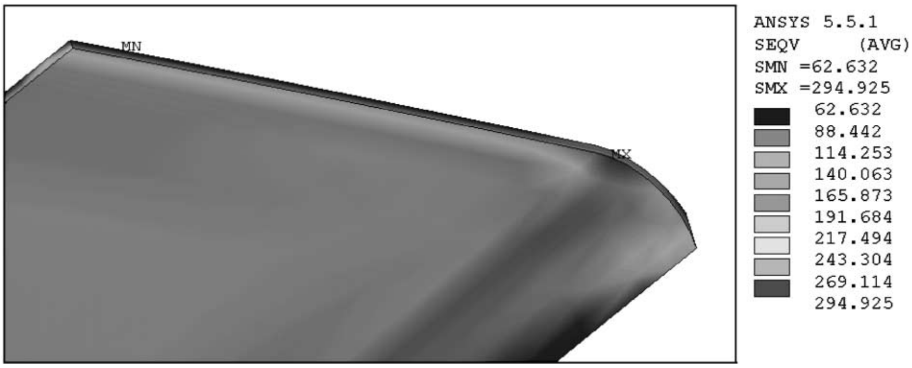

Fig. 6. Von Mises stress field in one corner of the SSM B_4C coating.

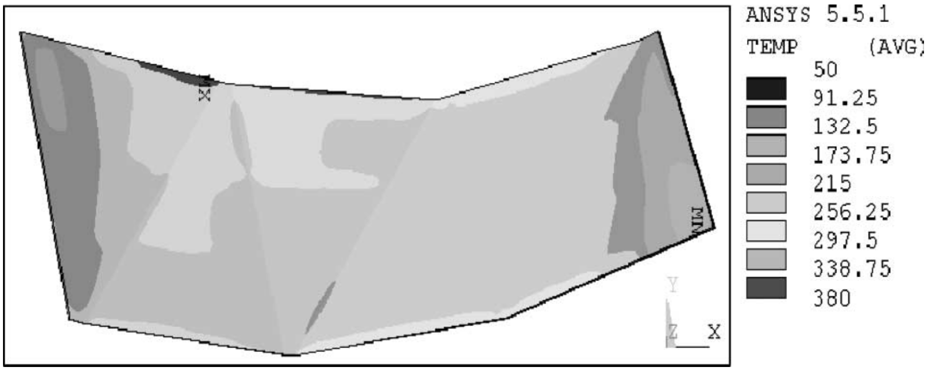

Fig. 7. Temperature field, in degree Celsius, on the FSM surface.

mum temperature in the FSM analysis is located at the plasma facing surface and is $\sim$ 360 °C (Fig. 7). Compared to the SSM, the 5 mm wall thickness, the higher cooling water temperature, the longer cooling water path, the higher heat load peak (600 kW/m^2) increase the peak surface temperature by up to 120 °C on the uncoated FSM. The deformation measurement showed that the dynamic water pressure (5 bar inlet, 2.5 bar outlet) does not contribute to the deformation of the mock-up. The numerical results are also in good agreement with the temperatures measured in different locations of the mock-ups during the experiments.

5. Conclusions

The thermo-mechanical tests and the characterization of boron carbide and silicon–boron–carbon coatings on stainless steel substrate showed that thick coatings, with thermo-mechanical and physical properties satisfying the W7-X requirements, are achievable. By the optimisation of the manufacturing parameters it has been possible to manufacture coatings with thermal conductivity above 2 W/m K, low porosity (VPS technique), and good adhesion/cohesion strength (>35 MPa) [8].

During the thermo-mechanical test, performed with the FIWATKA device, were applied heat loads up to 500 kW/m^2 on the coatings. This value is 2.5 times higher than the W7-X maximum operation first wall heat load that is 200 kW/m^2. After both steady state (from 50 to 500 kW/m^2 stepwise) and cyclic loading (1000 cycles at 500 kW/m^2 in 50 h) no failure was observed in the 500 μm thick B_4C and SIBOR coatings with interlayer.

References

[1] H. Bolt, Fus. Eng. Des. 22 (1993) 85.
[2] M. Sulik, H.P. Martinz, Plansee AG, Plansee leaflet 'SIBOR', 2000.
[3] G. Groboth, Thermophysikalische Eigenschaften von Schicht-Substrat-Verbunden – Mo mit Si(B,C) Schichten, Seibersdorf Report, ÖFZS-A-4328, 1998.
[4] H. Greuner, D. Glagla, O. Jandl, B. Mendelevitch, H. Renner, R. Rieck, Concepts and Prototype Elements of the Low-*Z* Wall Protection for the Stellarator W7-X, SOFT

2000 Conference and Proceedings, Madrid, September 2000.
[5] J.G. van der Laan, G. Schnedecker, E.V. van Osch, R. Duwe, J. Linke, J. Nucl. Mater. 211 (1994) 135.
[6] M. Lipa, E. Gauthier, Proc. 18th SOFT 1994, Fus. Tech. 1 (1995) 455.
[7] G. Schnedecker, Fusion Technology, Annual Report of the Association CEA/EURATOM' (compiled by P. Magaud), 1991.
[8] S. Kötterl, H. Bolt, H. Greuner, J. Linke, M. Mayer, V. Philipps, A. Pospieszczyk, H. Renner, M. Rödig, J. Roth, B. Schweer, D. Valenza, E. Vietzke, Development of Thick B_4C Coatings for the First Wall of W7-X, 9th International Workshop on Carbon Materials, Hohenkammer Castle, Germany, Sept. 2000.
[9] G. Hofmann, E. Eggert, The First Wall Test Facility FIWATKA, KfK 5381, 1994.
[10] W. Malléner, H.J. Groß, D. Stöver, Properties of Plasma Sprayed Boron Carbide Coatings, Proceedings of the 7th Thermal Spray Conference, Boston, Massachusetts, 20–24 June 1994, p. 627.