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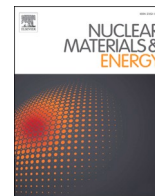
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Development of Boron Coatings for antenna protection limiters in the WEST Tokamak

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ABSTRACT

During the upgrade of Tore Supra to transform the machine into a fully metallic plasma facing environment (WEST), the Antennae Protection Limiters (APL), originally made in CFC, have been covered with a Tungsten coating with a Molybdenum interlayer (W/Mo/CFC).

However, due to the aging of the W/Mo/CFC coating, the APL have been redesigned and future antenna limiters will be made of copper alloy (CuCrZr) heat sink structure with a thick boron (B) coating to reduce remaining W impurity levels during plasma operations and address the blistering issues seen in previous coatings.

Among all the techniques able to realise B coating, Plasma Spray is the most appropriate technique owing to its simplicity, low cost and ability to deposit thick boron coatings on complex geometries.

Various boron coatings with thicknesses up to 900 μm were produced, characterized and their thermal behavior have been assessed using a specific laser heating program and a conventional electron/ion beam facility. This paper provides an overview of the R&D activities on boron coating developments and the related validation programs showing that the thermal conductivity of deposited films is drastically reduced compared to bulk boron, preventing use without additional coating optimization.

1. Introduction

During the upgrade of Tore Supra into WEST (W -for tungsten-Environment in Steady-state Tokamak), new plasma-facing components (PFCs) with tungsten surfaces have been developed to provide a full metallic environment. Beside the tungsten monoblock plasma facing units installed on the lower divertor for testing, other PFC have been covered with a tungsten coating to decrease the cost, manufacturing time and because the elements are located in less heavily loaded areas. In particular, the 10 Antennae Protection Limiters (APL) of the 5 antennae, made in CFCN11 (Carbon Fibre reinforced Composite) tiles brazed on CuCrZr heat sink structure have been covered with a tungsten coating with a Molybdenum interlayer (W/Mo/CFC) [1]. During the 5 experimental campaigns conducted between 2017 and 2020, WEST PFCs evolution has been monitored through visual inspections (the Articulated Inspection Arm has been used during the campaign and in-situ visual inspections have been done after each campaign). Due to the aging of the W/Mo/CFC coating (delamination, cracks) observed and the occurrence of blistering that causes arcs [2], it was decided to change the APL. In order to get rid of the carbon on the APL while enhancing the maintainability, APL have been redesigned into CuCrZr heat sink structure with a thick boron coating (Low Z) to reduce remaining W impurity levels during plasma operations and address the

blistering issues seen with W coatings.

This paper provides an overview of the R&D activities on Boron coating developments and the related validation programs. Finally, the article raises questions about the behaviour of boron coatings subjected to thermal flux and gives perspectives on the developments to be pursued.

2. WEST antenna protection limiters

WEST tokamak has 5 heating devices: 3 Ion Cyclotron Resonance Heating (ICRH) antennas and 2 Lower Hybrid Current Drive (LHCD) launchers. Antennae Protection Limiters (APL), located on both sides of antennae, protect each antenna from the plasma. APL are made of CFCN11 tiles brazed onto a water cooled support. During the upgrade of Tore supra into WEST, in order to provide a full metallic environment, and despite the fact that having high Z materials close to an ICRH antenna was known to increase the tungsten sputtering attributed to the acceleration of light impurity ions in the rectified RF sheaths [3], it was decided to cover the existing 10 APL with a double coating made of a first 80 μm molybdenum (Mo) layer followed by an 80 μm tungsten (W) coating prepared by Vacuum Plasma Spray (VPS) [1,4]. In order to operate long plasma discharges, infrared thermography systems monitor the 5 heating devices through real-time surface temperature

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measurements [5].

During several experimental campaigns, hot spots, caused by accelerated particles, have been observed by the infrared (IR) cameras on the APL of the LHCD antennas. After the campaigns, visual inspections revealed local damages on both LHCD and ICRH APL: coating delamination, blistering and CFC damage were observed [2] (Fig. 1).

To reduce remaining W impurity levels during plasma operations [3] and address the blistering issues seen in previous coatings, it was decided to redesign APL for CuCrZr heat sink structure covered with a low-Z material (Boron coating). APL must equip two types of antennas with different constraints: LHCD produces high and localized heat fluxes with fast electron beams while ICRH risk consists in high sputtering due to very energetic ions. In order to allow long pulse operation by addressing both LHCD and ICRH constraints, the redesigned APL is expected to be covered with thick boron coating. Initially, only 2 ICRH APL would be covered with Boron, allowing direct comparisons between antennas equipped with W or low-Z antenna limiters on WEST plasma. Then, the remaining antenna protections will be covered with the same coating.

The new APL are made up of 10 actively cooled copper alloy (CuCrZr) blocks with complex geometries (8 inner blocks and 2 outer blocks) assembled on stainless steel (Fig. 2).

3. Main boron coating specifications

One parameter that needs optimization is the coating thickness. While a substantial thickness of the B coating would be advantageous for extending the coating's lifespan, a thinner coating would improve the mechanical adhesion by reducing the interface stress. In order to find a compromise between coating longevity and coating integrity during WEST operation, various B thicknesses ranging from 100 μm to 900 μm have been compared. To ensure an homogenous component protection and thermal behavior, the coating has to be applied on the entire surface that could be in contact with the plasma (i.e. the plasma facing surface, the rounded edges and the lateral sides) and will be free of defect. Thickness non-uniformity will be minimized (range of $\pm 5 \mu\text{m}$ around the specified nominal B coating thickness) and a good adhesion of the coating must be ensured even after thermal stress (from high heat fluxes tests). In the context of plasma operation, the levels of impurities (especially C, O) will be minimized and kept below 50000 ppm and the minimal film density must be 90 % to ensure adequate thermal and mechanical properties of the coating.

4. Boron coating development and associated validation programs

Due to the need to apply thick B coatings, Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD) processes were not reasonably feasible. Consequently, thick boron coatings have been deposited by Vacuum Plasma Spray (VPS) process. ICB-PMDDM-LERMPMS laboratory of University of Technology of Belfort-Montbéliard has been chosen to proceed to B coating developments.

The coating development was divided in 3 steps:

- 1) First, the deposition processes were developed on small inertial CuCrZr samples (diameter 25 and 50 mm, thickness 10 mm) (Fig. 3a). The films were first characterized in terms of thickness, density and impurity levels, then the thermal behavior of the B coating thicknesses was established on the samples using two specific thermal cycles laser heating program leading to choose the optimal boron thickness.
- 2) The optimal coating thickness was then implemented on inertial representative mockup (120*100*70 mm) with chamfer and rounded edges (Fig. 3b) to assess coating uniformity and to evaluate the process ability to cover complex geometries.
- 3) Final optimized coating configuration was finally implemented on real size actively cooled APL block (Fig. 3c) and the performance of boron coating was assessed under cyclic high heat fluxes in the electron beam facility HADES.

4.1. VPS process development

In Vacuum Plasma Spraying (VPS), an electrical arc is generated within a low-pressure argon environment (600 mbar) to ensure that reactive gases such as oxygen and hydrogen do not disrupt the subsequent coating process.

Sifted grade P1 crystalline boron powder from the company Höganäs (Particles of sizes in the range of 14.4 μm -49.1 μm) are introduced into the plasma jet, where they undergo acceleration and melting. The plasma torch (F4-VB type from Oerlikon-Metco with a 6 mm diameter) is mounted on a mobile robotic arm, enabling uniform deposition on components with complex shapes. During the developments, an elutriation and classification of boron powder were necessary to remove all particles less than 10 μm and avoid problems at the powder injection point (the fine particles stick around the injection site, creating a clump which melts gradually and ejections of molten material may then be present on the deposit.) Each sample was instrumented with thermocouples to prevent overheating of the CuCrZr (<450 °C) which can lead

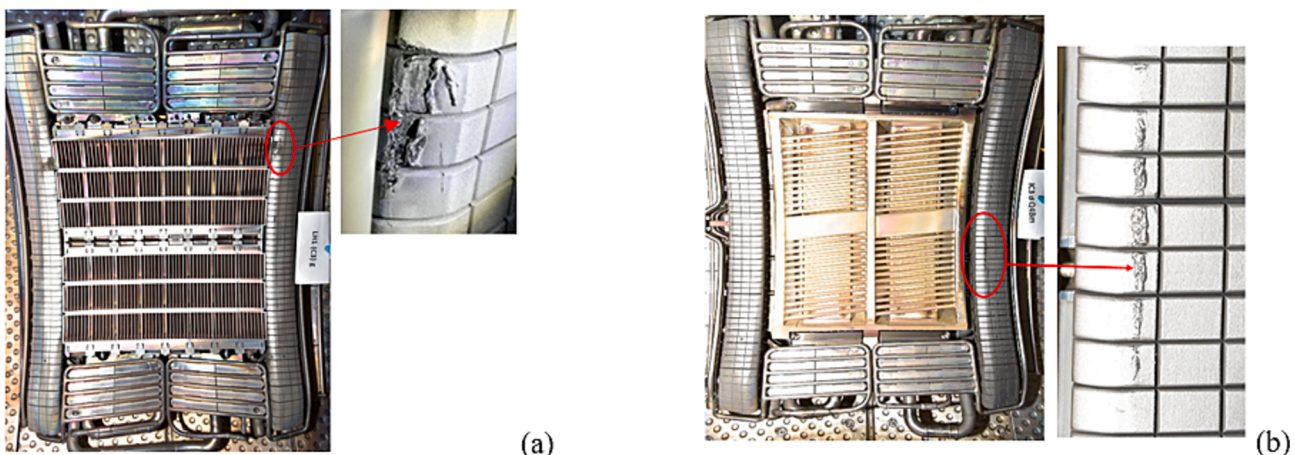


Fig. 1. W coating damages on (a) LHCD and (b) ICRH APL after the C4 campaign.

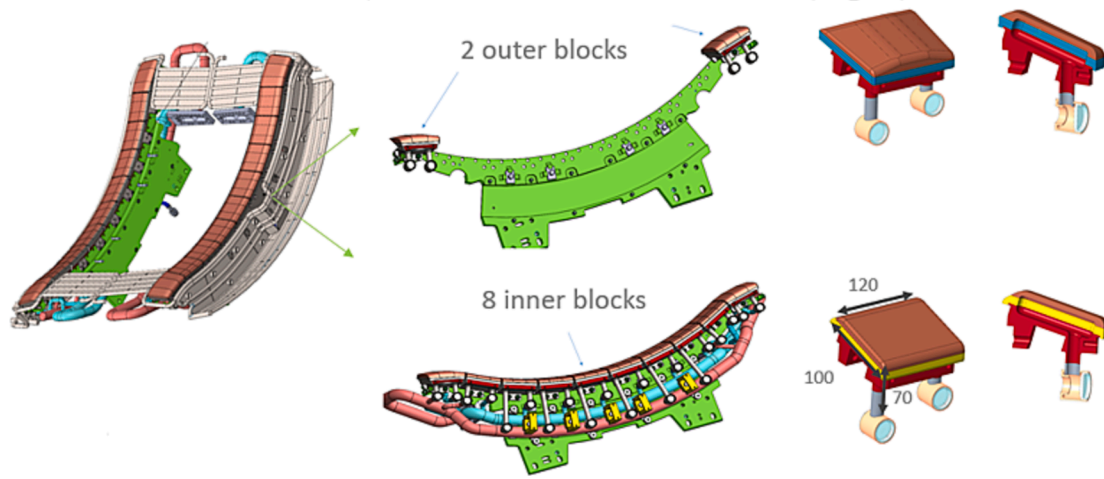


Fig. 2. Design of new ICRH APL – draft CAD model.

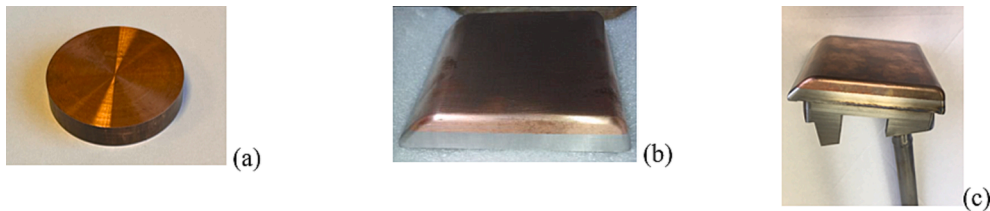


Fig. 3. Components used for B coating development: (a) CuCrZr sample (diameter 50 mm); (b) inertial mockup (120*100*70 mm) with chamfer and rounded edges; (c) real size actively cooled APL.

to significant degradation of its properties. It also prevents coating detachment caused by thermal mismatch between boron and CuCrZr at elevated temperatures. Different boron thicknesses were targeted (from 100 to 900 μm) to determine the influence of boron thickness on the adhesion of the deposit and adhesion measurements were carried out to explore whether residual stress phenomena might impose an upper limit on the maximum boron thickness.

From the first deposition tests, detachment of the coating was observed on 130 μm boron layers during the cooling phase (Fig. 4a). This detachment, associated to the difference in thermal expansion between CuCrZr substrate and boron deposit (respectively 17.10^{-6}K^{-1} and $7.2.10^{-6} \text{K}^{-1}$ at 25 °C) has been solved by introducing a 100 μm thick 316L interlayer (thermal expansion 16.10^{-6}K^{-1}) to mitigate the thermal expansion mismatch between B and the substrate (Fig. 4b).

4.2. Boron layer characterization

Adhesion tests (standard ASTM 633–69) performed on boron thicknesses from 100 to 900 μm (with 100 μm 316L interlayer) reveal that except for boron thickness of 100 μm where the rupture took place in the glue, the failures were predominantly observed within the boron layers.

This observation indicates that the particles making up the deposit lack cohesion between them, leading to the delamination of the boron layers (Fig. 5).

The density, assessed with SEM section, exceeds 92 % (some particles could be removed during sample polishing). (Fig. 6).

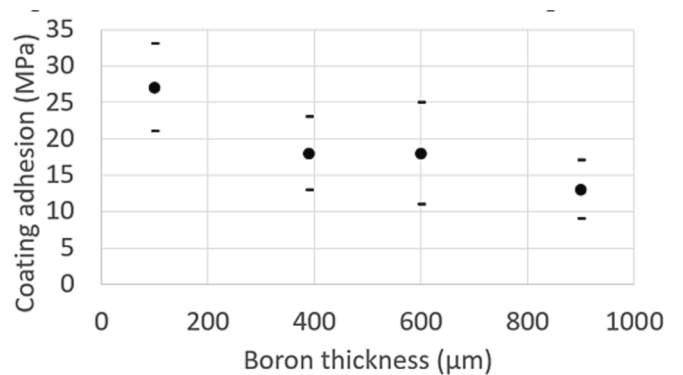


Fig. 5. Adhesion test (B + 100 μm 316L).



Fig. 4. (a) B(310 μm)/CuCrZr; (b) B(390 μm)/316L (100 μm)/ CuCrZr.

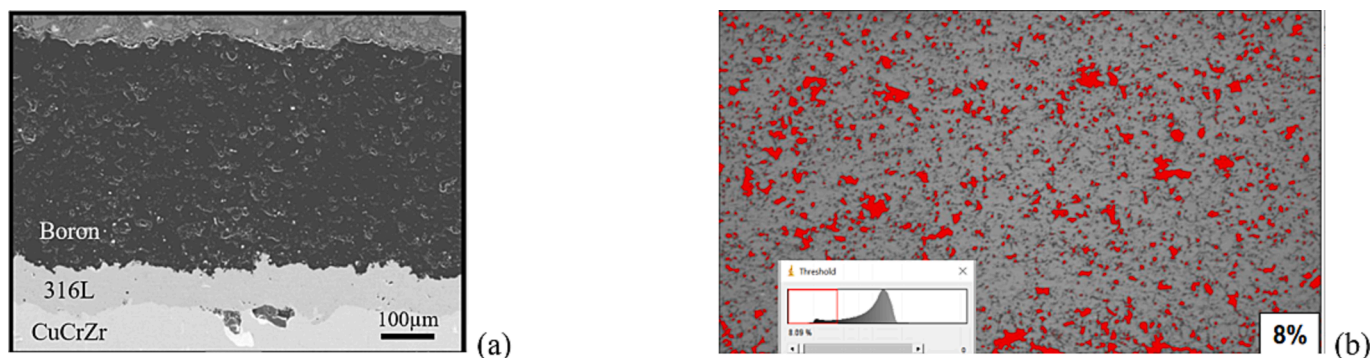


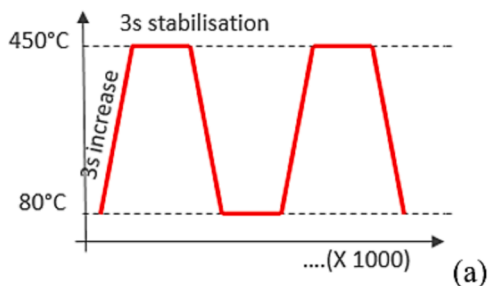
Fig. 6. (a) SEM view (cross section) B (290 μm)/316L(100 μm)/CuCrZr (b) density observation of boron layer.

Contamination of boron film, evaluated by CIA (Combustion Infrared absorption) for C, and RMIA (Reducing Melting Infrared Absorption) for O is: 99.08 % B; 0.38 % C; 0.44 % O.

4.3. Thermal behavior of B coating: Determination of optimal boron thickness

In order to determine the optimal boron thickness resulting in a defect-free film after thermal cycling, the thermal behavior of B coating thicknesses (140; 290 and 430 μm with 100 μm 316L interlayer) was established on inertial samples using a specific thermal cycle laser heating program. The experiments were conducted on the ChauCoLase platform (Chauffage Contrôlé par Laser / laser controlled heating) available at the Institut Fresnel [6,7]. This system utilizes a high power CW ytterbium fiber laser operating at 1080 nm that can deliver 1500 W of maximum power with a monomode laser beam and with a typical rising time of a few microseconds. The inertial sample was placed at the center of a low pressure vacuum chamber (10^{-2} mbar) on a water cooled support to maintain a CuCrZr temperature below 450 $^{\circ}\text{C}$. During heating, the power of the laser was regulated to sustain the temperature at a predefined setpoint. Real-time monitoring of the annealing process was carried out using an infrared camera while the sample temperature is regulated through a feedback loop that relies on optical pyrometry. The thermal performance of the coating during plasma operation is primarily influenced by its temperature and the temperature gradients within the coating itself. In that way, the laser beam diameter was set to 13.5 mm to minimize the pulse duration (and increase the temperature gradient) required to achieve a surface temperature of 450 $^{\circ}\text{C}$ (the pulse duration is 30 sec with a laser beam diameter of 50 mm and 3 sec with a laser beam diameter of 13.5 mm). Two sets of temperature up/down cycles were carried out:

- A first serie of 1000 cycles was performed by reaching a surface temperature of 450 $^{\circ}\text{C}$ (± 50 $^{\circ}\text{C}$), maintaining this temperature for 3 s,



stopping the beam, allowing the sample temperature to return to 80 $^{\circ}\text{C}$ and then repeating this cycle for a total of 1000 times (Fig. 7a).

The coating integrity was checked after the campaign and whatever the thickness of the boron (140; 290 and 430 μm), no delamination or defect have been observed.

- A second serie of thermal cycling was conducted until the boron coatings are damaged. For the 3 thicknesses of boron, a serie of 100 temperature rise/fall cycles were carried out. The temperature between each series was increased in steps of 50 $^{\circ}\text{C}$ until a degradation of the boron layer was observed (Fig. 7b). Unlike B coatings of 140 and 290 μm , cracks appear in the 430 μm B layer after thermal cycling at 700 $^{\circ}\text{C}$.

Based on these results, it was decided to continue developments with a boron thickness of 300 μm .

5. Validation on the boron coatings 300 μm thick (+100 μm 316L) on inertial representative mockup

A boron coating with 300 μm thickness (+100 μm 316L) was implemented on an inertial representative mockup (120*100*70 mm) with chamfer and rounded edges (Fig. 3b) to assess coating uniformity and to evaluate the process ability to cover complexes geometries. In addition to the delamination of the B coating observed on one edge of the mock-up (Fig. 8a), the thickness of the B coating was very heterogeneous over the surface of the component (evaluated by measuring the B thickness in different points of the inertial CuCrZr elements using optical microscopy profiles).

After polishing the edge to break the edge (too protruding), adjusting the deposition temperature, increasing the cooling duration between torch passes and adjusting the torch kinematics, the adhesion and homogeneity of the layer thickness were drastically improved (Fig. 8b). B layer covers the entire surface of the substrate (including the vertical portions and the rounded edges) with an acceptable B thickness dispersion.

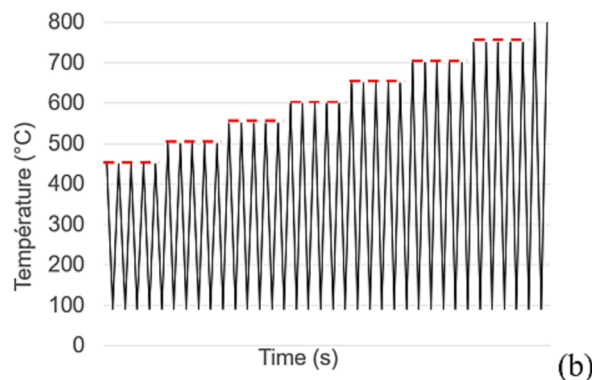


Fig. 7. Laser heating program conducted on the ChauCoLase platform: (a) 1000 temperature rise/fall cycles from 80 $^{\circ}\text{C}$ to 450 $^{\circ}\text{C}$ (b) 100 cycles temperature rise/fall cycles by increasing the temperature between each series in steps of 50 $^{\circ}\text{C}$ until a degradation of the boron layer.

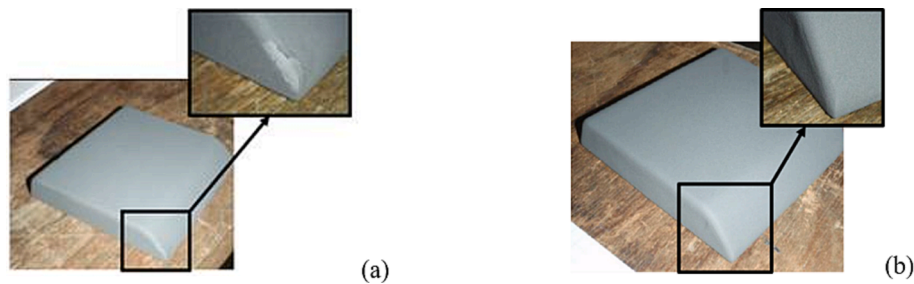


Fig. 8. (a) Non optimized B(300 μm)/316L(100 μm)/CuCrZr; (b) optimized B(300 μm)/316L(100 μm)/CuCrZr.

6. Validation on boron coated real size actively cooled APL and HHF tests in HADES

This final deposition process was finally implemented on real size actively cooled APL (Fig. 9) to assess the performance of boron coating under cyclic high heat fluxes in the electron beam facility HADES.

To test the thermal behavior of boron coated actively cooled mock-up, thermal cycling has been performed in the electron-beam facility HADES [8]. The surface temperature was monitored via an IR-camera and thermocouples have been placed in the hydraulic circuit in/out. The tests consist to apply repeatedly heat loads on the same area at a given level, in order to evaluate fatigue behavior of the coating. Finite element modelling was done using ANSYS, to determine the appropriate peak heat flux to reach the 450 °C limit. The Gaussian heat flux profile of the HADES beam with a central heat flux of 6 MW m⁻² resulted in a surface temperature of about 450 °C.

During the initial screening at 0.5 MW/m² (10sec duration; expected surface temperature 150 °C), an unusual temperature increase was observed in the mock-up, with temperatures ~ 295 °C (measured boron emissivity of 0.75), indicating an atypical thermal performance of the boron layers.

While the thermal conductivity of crystalline boron solids is extensively documented, there remains a notable lack of information regarding the thermal conductivity of amorphous boron films. Harikrishna and Golikova [9,10] reported that thermal conductivity of amorphous boron layer are significantly reduced relative to the values of 27 W/m.K reported for single and poly-crystalline B. It is also reported that the thermal conductivity of amorphous boron films clearly decreases as the mass density decreases [9]. It appears to be in the range of 0.9 to 2 W/m.K [9,10]. Using ANSYS simulations, the thermal conductivity of the coating layer is evaluated to 1.3 W/m.K (Fig. 10).

The tests were then stopped, as a heat flux of 6 MW/m² would have led to surface temperature in the range of 2200 °C.

7. Conclusion and perspectives

In the framework of upgrading WEST antennas APL into copper alloy



Fig. 9. Boron coated real size actively cooled APL.

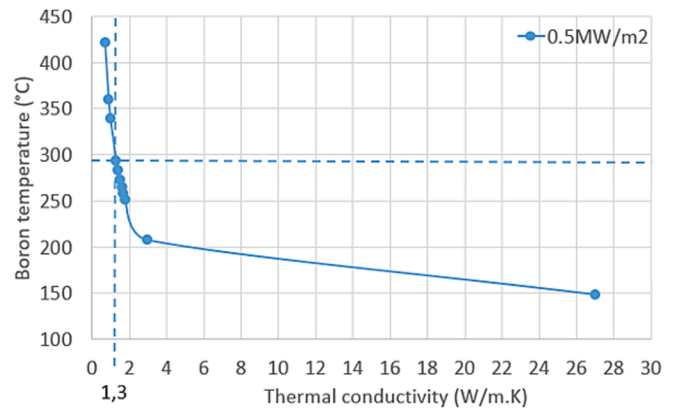


Fig. 10. Surface temperature of B coating (300 μm)/316L(100 μm)/CuCrZr exposed to a heat flux of 0.5 MW m⁻² (steady state) in function of boron thermal conductivity.

(CuCrZr) heat sink structure covered with a thick boron coating (Low Z), a boron deposition process compatible with complex shapes components has been developed. We characterized the films and improved the adhesion of the boron coating to CuCrZr by introducing a 316L (100 μm) interlayer and limiting the boron thickness to 300 μm to withstand thermal cycling without developing defects. The specifications were respected with a density greater than 92 % and a contamination level of less than 1 %. Assessment of B-layers thermal behaviour has been carried out using both a specific laser heating program and a conventional electron beam facility.

While during the laser heating program (where the power of the laser is regulated to sustain the temperature at a predefined setpoint), the thermal behavior of B coating was not influenced by B thermal conductivity, it is strongly influenced during HHF (High Heat Flux) tests where the heat load is fixed. In fact, it appears that an anomalous thermal behavior of the boron coating during HHF tests, as confirmed by ANSYS simulations, led to a boron thermal conductivities determined within the range of 1.3 W/m.K. This value was lower than the expected 27 W/m.K. We are currently in the process of measuring the thermal diffusivity of boron and 316L layers, and ongoing experiments are aimed at increasing boron density and reducing boron thickness.

CRedit authorship contribution statement

C. Hernandez: . M. Firdaouss: . A. Deleau: . M. Diez: . M. Misirlian: Supervision, Writing – original draft.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

References

- [1] M. Firdaouss, C. Desgranges, C. Hernandez, M. Richou, H. Greuner, B. Bös-wirth, I. Zacharie-Aubrun, T. Blay, J. Bucalossi, M. Missirlian, F. Samaille, E. Tsitrone, Overview of the different processes of tungsten coating implemented into WEST tokamak, *Fus. Engin. Design* 124 (2017) 207–210, <https://doi.org/10.1016/j.fusengdes.2017.02.087>.
- [2] M. Diez, Y. Corre, E. Delmas, N. Fedorczak, M. Firdaouss, A. Grosjean, J.P. Gunn, T. Loarer, M. Missirlian, M. Richou, E. Tsitrone, In situ observation of tungsten plasma-facing components after the first phase of operation of the WEST tokamak, *Nucl. Fusion* 61 (2021) 106011, <https://doi.org/10.1088/1741-4326/ac1dc6>.
- [3] V.V. Bobkov, F. Braun, R. Dux, A. Herrmann, L. Giannone, A. Kallenbach, A. Krivska, H.W. Müller, R. Neu, J.M. Noterdaeme, T. Pütterich, V. Rohde, J. Schweinzer, A. Sips, I. Zammuto, Assessment of compatibility of ICRF antenna operation with full W wall in ASDEX Upgrade, *Nucl. Fusion* 50 (2010) 035004, <https://doi.org/10.1088/0029-5515/50/3/035004>.
- [4] M. Firdaouss, C. Desgranges, C. Hernandez, C. Mateus, H. Maier, B. Bös-wirth, H. Greuner, F. Samaille, J. Bucalossi, M. Missirlian, Tungsten coating by ATC Plasma Spraying on CFC for WEST tokamak, *Phys. Scr.* T170 (2017) 014008, <https://doi.org/10.1088/1402-4896/aa8785>.
- [5] X. Courtois, M.H. Aumeunier, C. Balorin, J.B. Migozzi, M. Houry, K. Blanckaert, Y. Moudren, C. Pocheau, A. Saille, E. Hugot, M. Marcos, S. Vives, Full coverage infrared thermography diagnostic for WEST machine protection, *Fus. Eng. and Design* 146B (2019) 2015–2020, <https://doi.org/10.1016/j.fusengdes.2019.03.090>.
- [6] M. Minissale, A. Durif, G. Kermouche, M. Richou, L. Gallais, Grain growth, damages induced by transient heat loads on W, *Physica Scripta* 96 (2021) 124032, <https://doi.org/10.1088/1402-4896/ac27df>.
- [7] M. Minissale, A. Durif, P. Hiret, T. Vidal, J. Faucheux, M. Lenci, M. Mondon, G. Kermouche, Y. Pontillon, C. Grisolia, M. Richou, L. Gallais, A high power laser facility to conduct annealing tests at high temperature, *Rev. Sci. Instrum.* 91 (2020) 035102, <https://doi.org/10.1063/1.5133741>.
- [8] H. Roche, N. Vignal, A. Bureau, M. Missirlian, A. Martinez, T. Mondière, J. C. Hatchressian, V. Moncada, D. Mouyon, M. Richou, HADES - high heat load testing - facility at CEA-IRFM, *Fus. Eng. and Design* 192 (2023) 113769, <https://doi.org/10.1016/j.fusengdes.2023.113769>.
- [9] H. Harikrishna, W. A. Lanford, S. W. King, S. T. Huxtable, Thermal conductivity of plasma deposited amorphous hydrogenated boron, carbon rich thin films. *J. Nucl. Mater.* 514 (219), 154–160. <https://doi.org/10.1016/j.jnucmat.2018.11.031>.
- [10] O.A. Golikova, V.K. Zaitsev, V.M. Orlov, A.V. Petrov, L.S. Stilbans, E.N. Tkalenko, Conductivity of boron, of its crystal structure analoges, *Phys. Stat. Sol.* 21 (1974) 405–412, <https://doi.org/10.1002/pssa.2210210202>.