Project: “Synthesis, Characterization and Testing of Hydrogen Permeation Barriers (HPBs) applied as a safety measure for future fusion reactors”

Project type: Post-doctoral research

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Project acronym: SCTHPB

Summary of scientific and technical report,

September – December 2020

1. Summary

During the current reporting period, efficient permeation barriers of hydrogen isotopes in different components integrated in the fusion reactor were synthetized.

Numerous activities were held following the synthesis of permeation barriers from well-known suitable materials and also from different alloys which could express improved anti-permeation properties.

Thus, metallic, oxides and improved composites were realized using different plasma deposition techniques such as: TVA (Thermionic Vacuum Arc), AP-plasma (Atmospheric Pressure-Plasma Jet) and CMSII (Combined Magnetron Sputtering with Ion Implantation).

Numerous optimizations of the deposition parameters and substrate preparation were conducted in order to obtain high purity, compacted and adherent deposited layers. Supplementary, the HiPIMS method (High Power Impulse Magnetron Sputtering) was used in order to obtain high density metallic oxides depositions favored by the plasma in non-thermalized state. This method was successfully utilized as alternative to the CMSII method.

2. Scientific and technical report

2.1 Project management

Relevant activities were made in order to ensure the full logistic process such as the material acquisition (substrates), consumables (materials to be deposited), as well as the establishment of experimental configurations and manufacturing of deposited sample holders. Thus, utilized materials for the proposed deposition campaigns were acquired such as:

- oxides as targets (Al_2O_3, Cr_2O_3, Er_2O_3, SiO_2) for deposition by magnetron methods;
- powders with low granulation (<50μm) as W and Er_2O_3 for deposition by AP-plasma;
- austenitic steel 316L (low carbon), V, Zr and Al with different thicknesses and dimensions proposed as substrates.

In the official SCTHPB project website (http://scthpb.inflpr.ro/), the resulted deliverables are disseminated. This site contains relevant information which will be maintained up-to-date during the project duration.

2.2 Substrates preparation

Different materials such as SS (316L), V, Zr, Si and Al were prepared in order to improve the adherence for the deposited layer, in relation to the used method. For conducting permeation studies, the geometrical limitations imposed by the measuring facility were taken into consideration. The substrates need to have a diameter of 40 mm and a thickness of 0.5 mm, values indicated from the laboratory in Slovenia, were permeation measurements will take place. A precision cutting of the substrates was made utilizing an abrasive water jet (figure 1.a) with a nominal roughness of 1.6 µm (http://www.sidora.ro/date-tehnice.html).

(a) *(b)*

Figure. 1 (a) Final results of 316L SS sheet after water jet cutting; (b) sample support integrated in the AP-plasma deposition method;

In order to obtain an increased adhesion and in relation to the deposition method and to the substrate preparation procedure, substrates were acquired with the thickness of 0.5 respectively 2 mm.

AP-plasma method required a higher substrate roughness obtained by sanding technique and a preliminary test proved a minimum substrate thickness, arbitrary chosen at 2mm. In future permeation measurements, it will be taken into consideration this increase of substrate thickness which directly impacts the permeation measuring time and the overall contribution of the substrate to the measurements. The reference thickness deposited for the synthetized layers was established at 5µm.

2.3 Metallic layer synthesis

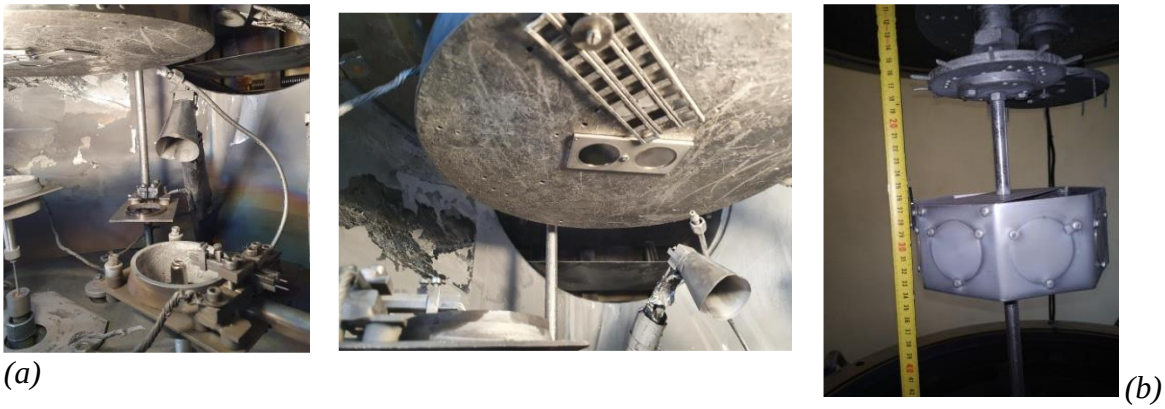
Here we report the W and Be depositions made by means of TVA method (figure 2.a), W depositions implying CMSII (figure 2.b) and AP-plasma methods.

With the TVA method, thin layers of W, Be and composite from W:Be (50:50 wt.%) (figure 2.a) were obtained. The applied parameters were:

- W- an arc voltage of 1.8kV, an arc current of 1.9A and a filament current with a mean value of 69.2A;

- Be – an arc voltage of 1.28kV, an arc current of 0.5A and the filament current with a mean value of 36.6A;

For both depositions, the used substrates were Al, SS and Si, Mo as deposition witness. For W+Be alloys we implied Al, SS, V and Zr. In order to avoid a possible layer delamination during or after deposition, the substrates were preheated at 100^{o}C.



(a) *(b)*

Figure 2(a) Deposition chamber interior of the TVA method with mounted substrates (pre-deposition); (b) Image from CMSII deposition chamber with mounted substrates (post-deposition).

Deposition of W by means of CMSII were held at a working pressure of $5x10^{-3}$, a magnetron current of 1.5A, with a total deposition time of 3.5 hours and a fluence of Ar (41 sccm). Additionally, we mention that the substrate polarization was set at a 100V and the deposited substrates were from Al, SS, V and Zr.

Depositions of W by means of AP-plasma method were conducted using W powder (99.95%, granulation of <44um) deposited on SS and Al, in the GTV 100kV facility (industry collaborator: https://plasmajet.ro/en/hardware/).

2.4 Oxides deposition synthesis:

Different types of oxides were deposited implying the use of AP-plasma, CMSII and HiPIMS.

The GTV 100kW facility was implied also for synthesis of the oxide layer. Each deposition was made at different parameters while keeping constant the distance between the substrate and the perpendicular material sputtering nozzle (110mm). The depositions were made on the 40 mm diameter disk with 2 mm thickness for ensuring the structural integrity during the sanding ablation process.

The parameters for 5µm deposition were:

- for Al_2O_3 (purity 99%) the deposition was made at a I=580A, voltage of 76V, with a pulverization rate of 38g/min and transportation gas with a fluence of 4l/min;

- for Al_2O_3 (60%) + TiO_2 (40%) alloy that was deposited as an alternative (at current of 650A and voltage of 67 V, Ar of 30l/min, H2 of 10l/min);
- Cr_2O_3 and Er_2O_3 were deposited with voltage of 74V, current of 360A, Ar fluence of 38l/min and debit h2-13l/min.

(a) *(b)* *(c)*

Figure 3. Deposited substrates example Ø 40mm/2mm from Al and SS with oxides deposited: Cr_2O_3 (a), Al_2O_3+TiO_2 (b), Al_2O_3 (c);

Using CMSII, tentative depositions were carried out for the oxides at the relevant parameters, but the duration of the stationary regime of plasma didn't facilitate the sputtering of the target, the cathode acting as an isolator.

Therefore, starting from the conventional deposition technique using magnetron discharge in DC (e.g. CMSII), in the deposition of oxides it was proposed to adopt an alternative technique as HiPIMS, which is a deposition method well-known in the literature for synthetizing more dense layers between (with 5-15%, comparing to CMSII or other DC magnetron sputtering based techniques) and more smooth due to the ionizing of sputtered material [*M. Samuelsson et al, doi.org/10.1016/j.surfcoat.2010.07.041*].

Therefore, implying HiPIMS method, the oxide layers (Al_2O_3, Cr_2O_3, Er_2O_3 and SiO_2) were realized in Ar atmosphere, with a constant fluence rate of 20 sccm, a gas pressure of 1Pa, a mean power of 100 W and a target-substrate distance of 10 cm. To maintain the discharge in the HiPIMS method, a negative voltage of 900V was applied, with the variable frequency between 1-4kHz, in pulsed and ultra-short regime proper for short-HiPIMS working conditions previously studied [*Velicu, Ioana-Laura et al, doi.org/10.1016/j.apsusc.2017.01.067*].

2.5 Synthesis of reinforced metal matrix composites

Metallic layers reinforced (W, Be) with oxides (Al_2O_3, Cr_2O_3, Er_2O_3 and SiO_2) deposited by TVA, AP-plasma and CMSII; Deposited oxides (Al_2O_3, Cr_2O_3, Er_2O_3 and SiO_2) by means of HiPIMS (at already mentioned parameters) were deposited simultaneously with W or Be, thus obtaining structurally enhanced layers by metallic addition.

Therefore, in the current reporting period, a number of 14 metallic layers, 16 metallic oxides and 8 metallic composites were synthetized (as mentioned in below table).

Used method: Metallic single layers / (substrates)	**Used method:** Metallic oxides / (substrates)	**Used method:** Matrix composites / (substrates)
TVA: • W / (Al, SS); • Be / (Al, SS); • W:Be / (Al, SS, V, Zr); **CMSII**: • W / (Al, SS, V, Zr); **AP-Plasma**: • W / (Al, SS);	**AP-plasma**: • Cr_2O_3 / (Al, SS); • Er_2O_3 / (Al, SS); • Al_2O_3 / (Al, SS); • Al2O3+TiO2 / (Al, SS); **HiPIMS**: • SiO_2 / (Al, SS); • Cr_2O_3 / (Al, SS); • Al_2O_3 / (Al, SS); • Er_2O_3 / (Al, SS)	**HiPIMS**: • W / SiO_2(Al); • W / Cr_2O_3(Al); • W / Al_2O_3(Al); • W / Er_2O_3(Al); • Be / SiO_2(Al); • Be / Cr_2O_3(Al); • Be / Al_2O_3(Al); • Be / Er_2O_3(Al).

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