ON TUNGSTEN SPRAYING USING INDUCTIVELY COUPLED PLASMA SYSTEM - FIRST RESULTS

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Abstract

Thanks to its unique properties such as high melting point and density, tungsten and tungsten-based alloys are commonly used in a wide range of applications. Among others, these materials are promising candidates for the plasma facing components in the future fusion reactors. One of considered ways of production of these parts is plasma spraying. There are however several limitations for conventional gas stabilized torches, mainly in plasma enthalpy (i.e. the ability to efficiently melt tungsten particles in considerable feed rates) and susceptibility of tungsten to oxidation (which complicates spraying in oxidizing open-air atmosphere). The radio frequency inductively coupled plasma torch (RF-ICP) is a unique system which can potentially overcome both these problems and can be used for efficient tungsten spraying. The tungsten powder, which can be finer than the one used for the conventional systems, is fed axially into the hot plasma core; both factors lead to a more efficient melting of the particles. The deposition is performed in a chamber with controlled atmosphere of inert gas or decreased pressure, the oxidation is therefore suppressed. In this first study carried out with the newly commissioned RF-ICP system TekSpray 15 (Tekna), samples of tungsten coatings on graphite substrates were prepared. The X-ray diffraction and SEM images of the free surfaces and cross-sections were obtained, documenting high purity of the deposits and appropriate flattening of the splats leading to a dense coating microstructure. The effect of substrate preheating on the microstructure, porosity and hardness was also studied.

Keywords: Radio frequency inductively coupled plasma torch, tungsten, plasma facing components, plasma spraying

1. INTRODUCTION

Majority of the unresolved issues concerning the design of future nuclear fusion reactor is related to materials. The reactor parts referred as first wall and divertor (the so called plasma facing components or PFCs) will be subjected to harsh conditions that will gradually deteriorate properties of the wall material. In fusion environment, the PFCs will be subjected to a complex loading involving high fluxes of heat and particles from the fusion plasma, associated mechanical loads, neutron irradiation, etc. [1]. Very few materials are suitable for such conditions, and these are subject of worldwide research and development. For the ITER PFCs, tungsten and beryllium are planned, while for the next step device, DEMO, tungsten and tungsten alloys are foreseen as the primary candidates. Advantageous properties of tungsten-based materials include high melting point, good thermal conductivity, high resistance to sputtering and low tritium retention. Among tungsten's drawbacks are difficult machining, intrinsic brittleness at lower temperatures and propensity to recrystallization at higher temperatures.

Plasma spraying is an alternative technology for the production of the PFCs. Generally, plasma spraying involves melting of a material in form of a powder and its deposition on another bulk material frequently called substrate. In the terms of advanced PFCs, the deposited material is tungsten and the substrate is a structural material, e.g. copper or special steel grades. Plasma spraying has several advantages - namely being a single-step manufacturing technology, without the need for further joining, the ability to coat large-area components, including non-planar surfaces, easy formation of graded composites, relatively low heat input to the coated parts, relatively low cost and high coating thickness capability [2]. The major disadvantage of the plasma sprayed coatings, stemming from their layered and porous structure, is their relatively low thermal conductivity. Oxidation also significantly contributes to the unfavourable properties since oxides are located mainly at the interlayer boundaries, thus further lowering coating cohesion and thermal conductivity. Coating properties can be improved by suppressing oxidation during the deposition process which can be realized by various ways, e.g. chemically by addition of WC to the deposited powder [2], by application of various shrouding chambers [3] or by deposition in vacuum [4]. The induction coupled plasma (ICP) method is a unique plasma spraying technology that involves deposition of the coatings in a reactor chamber. For the purpose of tungsten coating production, the chamber can be either evacuated of filled with inert or reducing gas.

In this first study carried out with a newly commissioned RF-ICP system, samples of tungsten coatings on graphite substrates were prepared. Besides X-ray diffraction analysis and SEM observation of the coatings, the effect of substrate preheating on the microstructure, porosity and hardness was also studied.

2. EXPERIMENTAL

Two samples of tungsten (W) coating on a graphite substrate were prepared with the RF-ICP system TekSpray 15 (Tekna). Mean particle size of the used powder was under 63 µm and the powder was fed axially into the plasma core, approximately in the middle of height of the coil inducing the plasma. The feed rate was around 13 $g \cdot min^{-1}$, the spraying distance, measured from the end of the torch, was 70 mm. The mixture of argon and hydrogen gas was used as plasma-forming gas and a slight overpressure around 1034 kPa was kept in the chamber during the deposition. The substrates were disc-shaped, 6 cm in diameter and 1 cm in height and rotated during the spraying with the speed of 22 RPM. The disc center was shifted 5 mm from the axis of the plasma jet in order to avoid local overheating of the substrate center. The powder was sprayed on both substrates for one minute, however one substrate was preheated in the plasma jet (with deactivated powder feeding) for one minute before the deposition, while the other was not. The appearance of the samples after the spraying with the deposited tungsten coating is illustrated in **Figure 1**. The specimens were cut in half, mounted in resin and metallographic cross-sections were prepared. The samples were grinded on SiC paper up to grit size FEPA 4000 and polished with 1 µm polycrystalline diamond suspension for 40 min on the machine Tegramin-25 (Struers). The last step of the preparation was 20 min of the oxide polishing with 5:1 mixture of OP-S suspension and 30 % H_2O_2

Phase composition of the as-sprayed samples was investigated on the free surface by X-ray diffraction method (XRD). Diffractometer D8 Discover (Bruker) assembled in Bragg-Brentano symmetric geometry was equipped with polycapilary optics creating a parallel beam of 1 mm diameter and 1D detector LynxEye. Scanning electron microscopy (SEM) of free surfaces and cross-sections of the samples was performed on EVO MA 15 (Carl Zeiss), the elemental analysis was performed by energy-dispersive X-ray spectroscopy (EDS) on EDS Quantax (Bruker). The porosity of the coatings was evaluated by image analysis of the cross-sections images, the hardness was measured on the Nexus 4504 (Innovatest) with the load of 0.3 kgf.

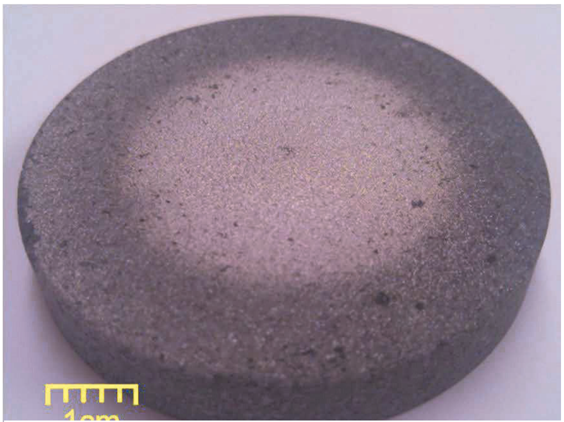

Figure 1 Preheated sample after tungsten coating deposition

3. RESULTS AND DISCUSSION

Coatings with metallic appearance (**Figure 1**) were successfully deposited in both experiments. X-ray diffraction patterns obtained from the free surface of the samples are presented in **Figure 2** and confirmed, that a very pure W coating was obtained. The EDS analysis from the cross-sections discovered besides tungsten also small amount of oxygen (around 1 wt. %) but no oxidic splats were observed within the coating microstructure. Detection of oxygen might have been caused by the cross-section preparation process. Nevertheless, these results are very promising as similar oxygen contents were obtained with the water stabilized plasma torch only with use of a special shrouding chamber and after thorough optimization of spraying conditions [3].

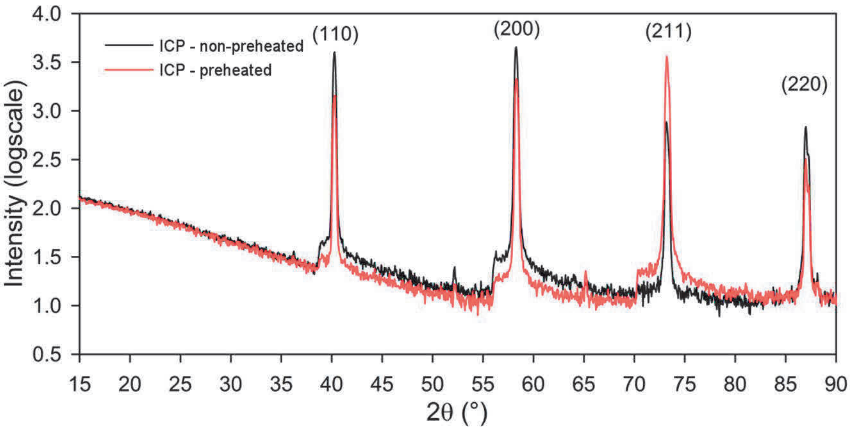

Figure 2 Diffraction patterns from the free surfaces of the samples documenting pure W coating

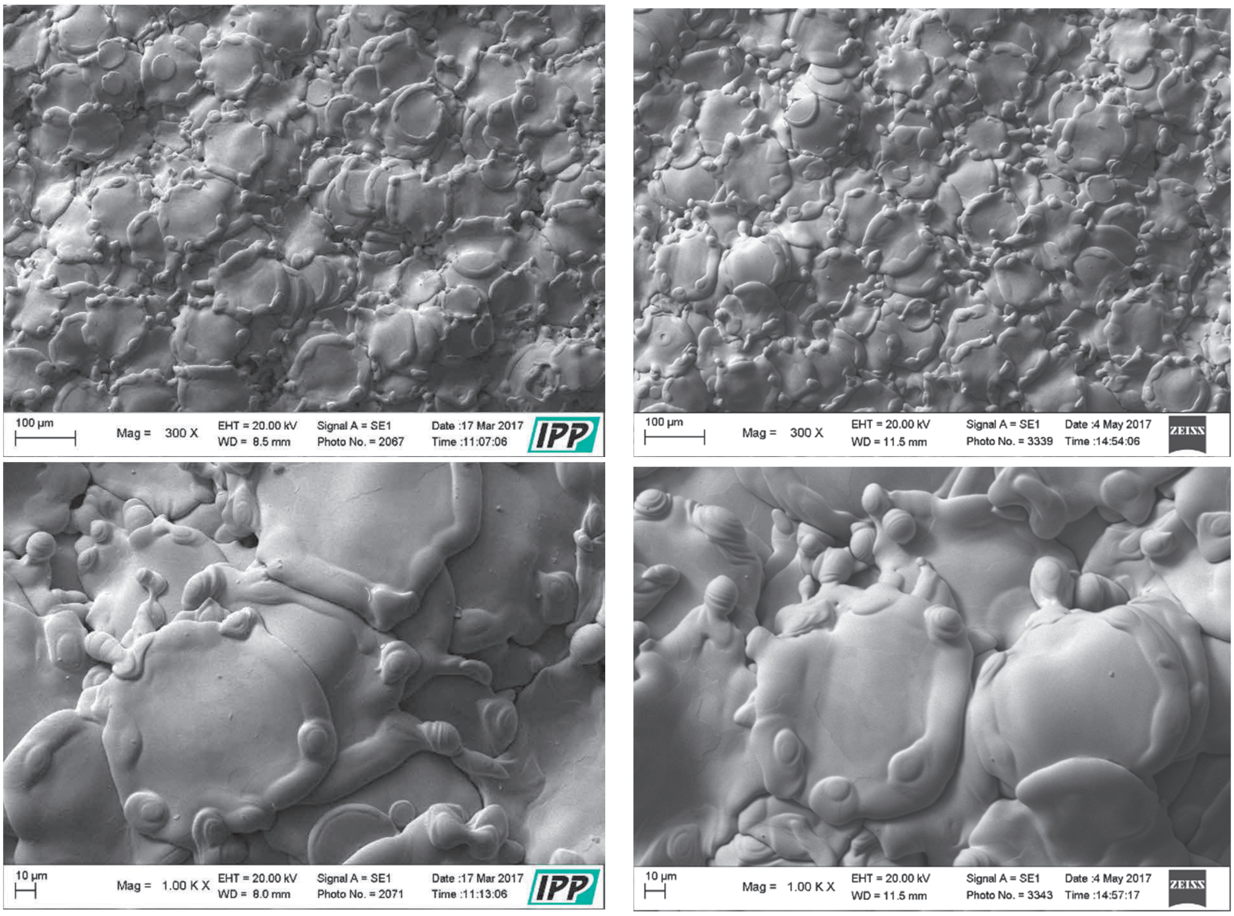

Figure 3 Micrographs of the coatings free surface: without preheating (left) and with preheating (right)

The micrographs of the coating free surfaces are presented in **Figure 3**. Both samples exhibit formation of appropriate pancake-like splats which suggests also good melting of the powder during the spraying.

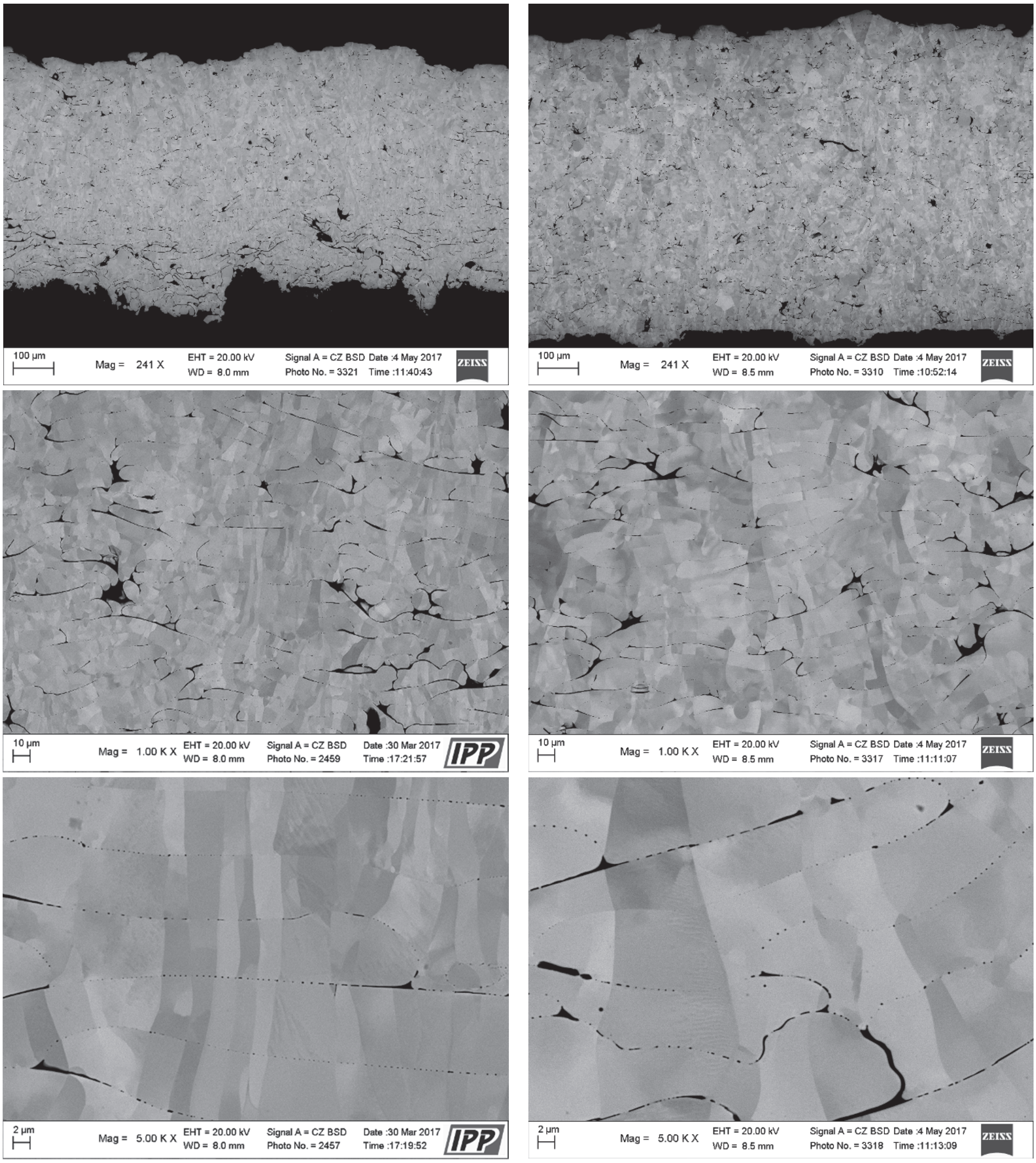

Figure 4 Microstructure of the coatings: without preheating (left) and with preheating (right), general overview (top row), cross-section showing span of the columnar grain trough several splats (middle row), detail of the columnar grains (bottom row)

The microstructures of the cross-sections are presented in **Figure 4**. The maximum thickness of the coating was around 570 µm for the sample without preheating and around 730 µm for the preheated specimen. This difference suggests that the preheating might have an influence on the deposition efficiency. This hypothesis however has to be statistically validated. The main difference in the appearance for the two samples is in the porosity of the coating close to the substrate which is obviously slightly higher for the sample without preheating. Once the sample was heated enough during the course of spraying, the microstructure started to be very similar to the one of the preheated sample. The outer layers of the cross-section (and also surfaces presented in **Figure 3**) therefore look very similar for both samples. Sensitivity of the porosity of sprayed tungsten to the preheating was observed also in [5].

Despite porosity gradient of the non-preheated sample, the mean porosity of the deposited coatings was between 3 - 4 % which is also a very promising for further experiments. The values of Vickers hardness HV0.3 were measured in different depths of the coatings and did not show any significant difference, since the hardness values were practically identical, i.e. 370 ± 32 HV0.3 for the non-preheated and 370 ± 31 HV0.3 for the preheated specimen. Substantial deformation of the deposited tungsten under the indent is apparent from **Figure 5**.

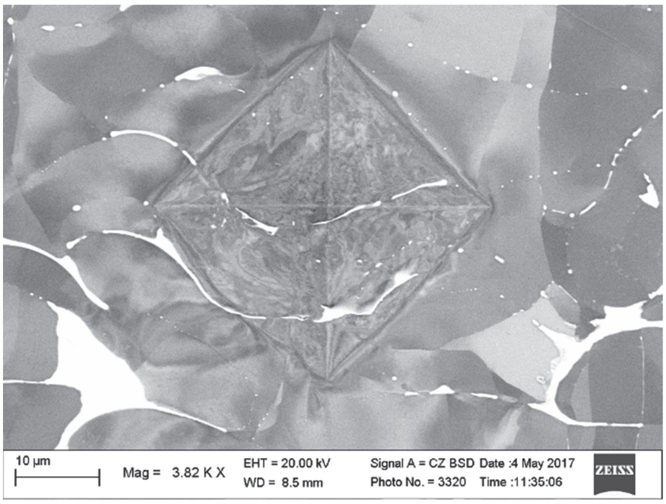

Figure 5 Indentation in the cross-section of the preheated sample

4. CONCLUSION

Two samples of tungsten coating on graphite substrate were prepared with RF-ICP plasma torch TekSpray 15. The influence of the substrate preheating on the microstructure, porosity and hardness was evaluated.

First tungsten coatings deposited by RF-ICP show promising quality. The coating was compact with relatively low porosity (3-4 %) and showed appropriate formation of splats. The oxidation was suppressed during the spraying, though low amount of oxygen (around 1 wt. %) was detected in the coating. Overall, the RF-ICP seems to be promising technology for production of parts made of tungsten and tungsten based alloys, such as plasma facing components. The first comparison of spraying on the preheated and non-preheated substrate shows differences in porosity of first sprayed layers, the hardness on the other hand did not show any significant difference. Other measurements (e.g. thermal conductivity) need to be performed and effect of other spraying parameters on the resulting coating will be evaluated.

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REFERENCES

[1] MATĚJÍČEK, J., WEINZETTL, V., DUFKOVÁ, E., PIFFL, V., PEŘINA, V. Plasma Sprayed Tungsten-based Coatings and their Usage in Edge Plasma Region of Tokamaks. *Acta Technica CSAV,* 2006, vol. 51, no. 2, pp. 179-191.

[2] MATĚJÍČEK, J., CHRÁSKA, P., LINKE, J. Thermal Spray Coatings for Fusion Applications-Review. *Journal of Thermal Spray Technology*, 2007, vol. 16, no. 1, pp. 64-83.

[3] MATĚJÍČEK, J., VILÉMOVÁ, M., NEVRLÁ, B., KOCMANOVÁ, L., VEVERKA, J., HALASOVÁ, M., HADRABA, H.. The influence of substrate temperature and spraying distance on the properties of plasma sprayed tungsten and steel coatings deposited in a shrouding chamber. *Surface and Coatings Technology*, Available online 22 October 2016.

[4] XIAOYONG, Z., LAIMA, L., PING, L., JUNLING, C., GUANGNAN, L., YUCHENG, W., Thermal Performance and Microstructure of Vacuum Plasma Sprayed Tungsten Coatings under Cyclic Heat Load. *Rare Metal Materials and Engineering,* 2015, vol. 44, no. 10, pp. 2405-2408.

[5] KOVÁŘÍK, O., HAUŠILD, P., SIEGL, J., CHRÁSKA, T., MATĚJÍČEK, J., PALA, Z., BOULOS, M. The influence of substrate temperature on properties of APS and VPS W coatings. *Surface and Coatings Technology,* 2015, vol. 268, pp.7-14.