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High heat load testing of plasma sprayed W coatings

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Abstract

The influence of five spraying parameters on the thermal shockresistance of plasma sprayed tungsten coatings was evaluated with a pulsed electron beam gun. The pulse duration was 0.2 s and the absorbed power density 60 MW/m^2. Two series of samples were tested. Both were plasma sprayed in controlled inert atmosphere, one at atmospheric pressure (AP) and the other at low pressure (LP). The porosity seems to be a positive factor for thermal shock resistance: the cracks are more numerous and thinner in less dense specimens. Moreover, the coating thickness is a crucial factor. Indeed, the 100 μm thick coatings (LP and AP) showed no delamination whereas 1 mm thick AP coatings suffered edge delamination.

Keywords: Tokamak de Varennes; High Z wall material; Physical erosion; Wall coating

1. Introduction

The technique of plasma spraying which may be highly automated and robotized is currently under investigation as a coating technique to protect and repair surfaces exposed to the plasma in fusion reactors [1,2]. The divertor plates of ITER will have to sustain, in normal operation, heat fluxes of the order of 10 MW/m^2 and will be eroded at a rate of 5 mm a year (for tungsten) [2]. Moreover, during plasma disruptions, the heat load can reach 10 MJ/m^2 deposited in 0.1 ms, leading to severe localized damage of the divertor plates. Thus the possibility of repairing the exposed parts in the reactor using plasma spraying is an attractive solution to this tremendous materials problem.

High melting point metals such as tungsten and its alloys are currently considered as potential candidates for the plasma-exposed divertor surfaces [1,2]. They have a low sputtering yield and are not subject to chemical erosion by hydrogen such as carbon-based materials. One of the main disadvantages of these high Z materials is the important radiation energy loss associated with the presence of partially ionized impurities of these materials in the plasma. However, the ionization distance in the plasma is shorter for tungsten than for lighter elements, favoring a rapid redeposition of the sputtered atoms on the divertor plates, and thus low plasma contamination by these impurities [3].

The coatings produced by plasma spraying are built by successive accumulation of molten or partially molten droplets spreading on the substrate surface and forming thin lamellae. The thermal contact between these lamellae is not, in general, perfect and is limited by the presence of thin pores or secondary phases (e.g., oxides and nitrides) at the interface between lamellae [4]. In previous papers the influence of five spraying parameters on the microstructure [5] and the related thermal diffusivity [6] of tungsten coatings has been studied. It was shown that the nature of the surrounding atmosphere is the principal spraying parameter and that the percentage of good contact between lamellae is a crucial structural factor influencing the diffusivity of the sprayed coatings. In the case of low pressure plasma sprayed (LP) tungsten coatings [7] the thermal conductivity was approximately 60% of the value for high-purity bulk tungsten. For atmospheric pressure plasma spraying this value depends on the nature of the gas; it is about 5% in air, 15% in nitrogen [6] and it has recently been increased to 35% in argon [8].

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Table 1
Spraying conditions for AP coupons

Spraying conditions	A	B
Powder	SP12430 (−75 + 30 μm)	Amperit 140.3 (−45 + 5.6 μm).
Spraying distance	75 mm	120 mm
Arc gas	Ar 50 l/min + He 24 l/min	Ar 50 l/min + H_2 2.7 l/min
Thickness	100 μm	1 mm
Spraying atmosphere	argon	nitrogen

The thermal shock resistance of refractory coatings is affected by many factors. Refractory coatings (TiC, W, ...) are often brittle and difficulties may be encountered with their adherence to the substrate. Some of these factors are related to the nature of the shocks (intensity and frequency) while others refer to the properties of the coatings: the difference in thermal expansion coefficient between the substrate and the coating, the substrate surface preparation before coating deposition, the thickness of the coating, the presence of microcracks which can be perpendicular or parallel to the interface, and the porosity. In the case of TiC coatings [9], tests made in TdeV have indicated that thin coatings are more resistant to thermal shock but do not ensure sufficient protection to the substrate which can be locally melted. As the thickness is increased, an optimum situation is attained where only a network of microcracks perpendicular to the interface are observed (segmentation of the surface). For thicker coatings, delamination near the coating–substrate interface occurs. The optimum thickness can be increased by substrate surface preparation (ex. grooves) [9].

LP tungsten has been tested in the electron beam test facility of Sandia National Laboratory, Albuquerque [10] under long pulses (up to 60 s) with cooling period between pulses. The sprayed coupons showed stable performance at power levels up to 16 MW/m^2. This paper reports preliminary results on the thermal shock resistance of plasma sprayed tungsten under short (0.2 s) pulses at a higher power level (60 MW/m^2). Coatings were prepared varying five spraying parameters: the nature of the atmosphere, the thickness of the coatings, the plasma arc gas, the powder size and the spraying distance.

2. Experimental procedure

2.1. Spraying conditions

A first series of coatings were sprayed in a controlled atmosphere (AP- with argon or nitrogen) using a SG100 Miller gun (129-145-130). The coatings were deposited on grit blasted coupons (20 × 50 × 7 mm) of TZM molybdenum alloy. On each coupon two regions (15 × 22 mm) were coated. Two types of powder, two spraying distances, two arc gases and two spraying atmospheres were used. These spraying conditions are given in Table 1. The powder feed rate was about 45 g/min. The plasma power was 27 kW with the Ar + He plasma gas mixture. With the Ar + H_2 mixture, the power levels were 30 kW and 26 kW when spraying the coarse powder (SP 12430) and the fine one (Amperit 140.3), respectively. Table 2 gives for each coupon the specific level of the five factors. A second series of coatings were deposited under proprietary conditions under low pressure (LP) on TZM coupons with a fine powder. The resulting coatings are somewhat denser but the density varies with the spraying conditions. Two LP coupons were prepared (22-1 and 22-2) with a thickness of about 100 μm.

2.2. Thermal shock resistance

The thermal shock resistance has been evaluated with an electron beam gun operated at 10 kV and 0.8 A on a spot of about 0.8 cm^2. The incident power density obtained is about 100 MW/m^2. Only about 60% of this energy is actually absorbed in the coating due to backscattered electrons [11]. So the absorbed power density is about 60 MW/m^2 (much more severe than the expected level in normal operation in divertor). As mentioned above, two regions were sprayed on each coupon prepared according to conditions given in Table 2. Region α and β were submitted to a series of 100 shocks lasting 0.2 s and region β to 100 shocks lasting 0.5 s. The samples were examined by SEM after 40 and 100 shocks as well as before the tests. A video camera was used to determine the onset of

Table 2
Design matrix for AP coupons

Coupon	Powder	Spraying distance	Arc gas	Thickness	Spraying atmosphere
2	A	B	A	B	A
3	A	B	B	A	A
4	A	B	B	B	A
7	B	A	B	A	A
8	B	A	B	B	A
9	A	A	A	B	B
10	A	B	A	B	B

delamination if any. A thermocouple was in contact with the back face of the substrate and the maximum temperature recorded after 40 shocks was about 550°C (and after 100 shocks the maximum was 650°C).

3. Experimental results and discussion

3.1. Microstructure of coatings before thermal shocks

The cross-section aspect of AP and LP samples is shown on Fig. 1. The variation in density between the two LP samples is illustrated in Fig. 1a, b. The coatings sprayed at atmospheric pressure (AP samples 3 and 7), show very different structures depending on the size of the tungsten powder used. Sample 3 (Fig. 1c) sprayed with the coarser powder has thicker lamellae and larger pores than those observed in sample 7 (Fig. 1d) sprayed with the fine powder.

3.2. Thin coatings (AP and LP) after thermal shocks

The cracks observed after the thermal shocks are significantly wider in the denser LP coating. Fig. 2a, b show that sample 22-2 is less dense than sample 22-1 and has much thinner cracks. In the case of sample 22-1 the cracks width is about 10 μm near the coating surface making them easily visible from the surface under optical microscopy. In both cases the cracks propagated across the complete coating thickness. The aspect of the surface of the samples is shown on Fig. 3. The influence of the powder size is illustrated. The LP samples have been sprayed with a very fine powder as it appears on Fig. 3a. On the contrary the very smooth aspect of the 'pancakes' visible on Fig. 3c is caused by the spraying of the coarse powder used for some AP coatings. The finer powder projected for the other AP coatings (Fig. 3b) produced an intermediate topography. In the LP samples, the cracks after 40 shocks are long and not numerous. Inversely in the fine powder AP sample (Fig. 3b) a fine mesh network of cracks is formed during the shocks.

3.3. Thick coatings (AP) after thermal shocks

All thick coatings suffered edge delamination in test conditions used in this study, as well as cracking, as observed in thinner coatings. The cracks are relatively

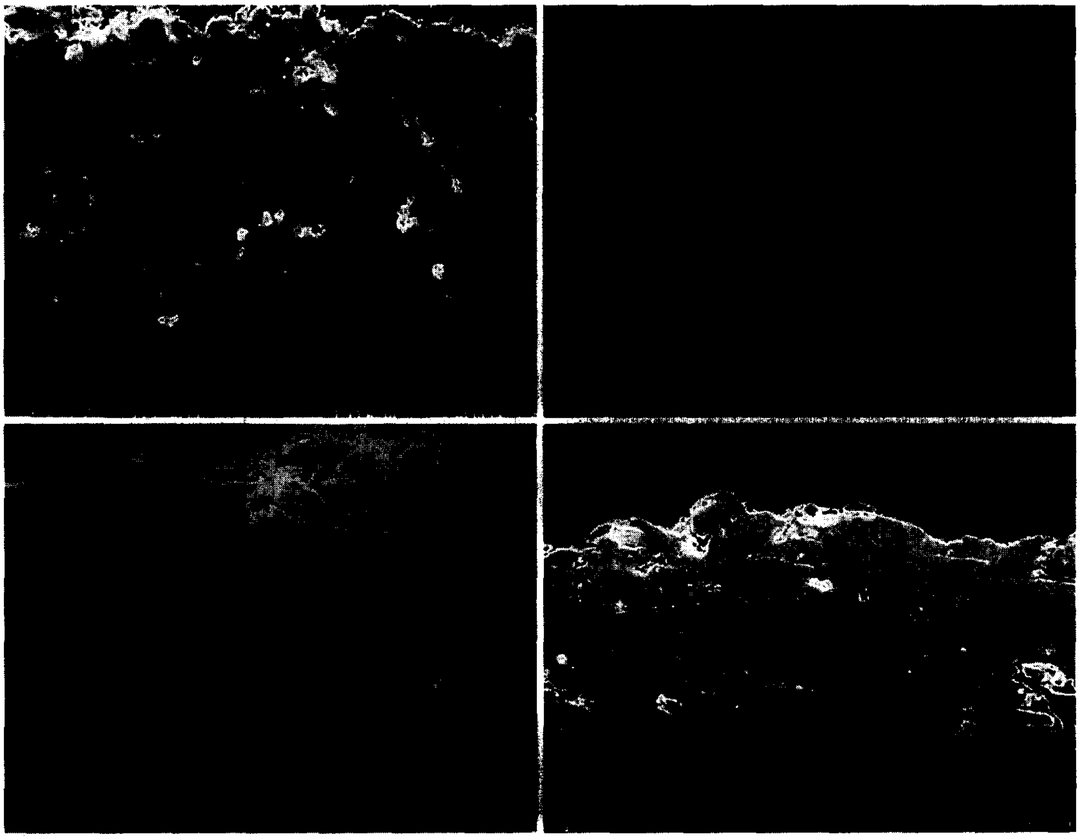

Fig. 1. Cross-section SEM micrographs of LP coatings (a: 22-1 and b: 22-2) and AP coatings (c: 3 and d: 7).

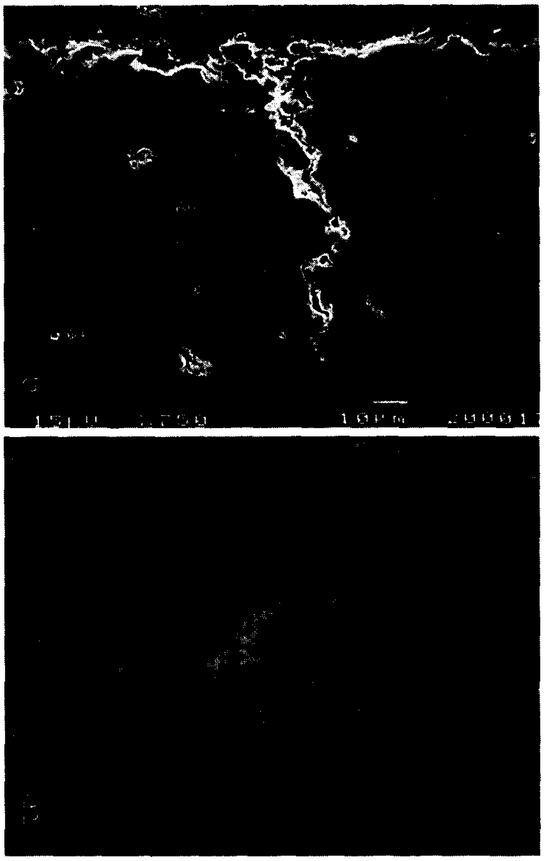

Fig. 2. Cross-section SEM micrographs of LP coatings after 40 shocks (a: 22-1 and b: 22-2).

wide, as indicated in Table 3. Sample 10β resisted longer; the edge delamination progressed slowly after each shock, as observed with the video camera. Sample 10α showed no delamination after 100 shocks.

If one compares coatings 9 and 10 (the most resistant thick coating) they vary only by the spraying distance; both are sprayed in nitrogen and 9 is sprayed with a spraying distance of 75 mm. Coatings 2 and 10 differ by the spraying atmosphere, 2 being sprayed in argon. As observed experimentally, the substrate reaches a higher temperature during spraying in argon. Using a short spraying distance has the same effect. This produces a coating with better interlamellar contact [5]. The net result will be twofold: (a) the coating will have a better thermal conductivity [6] and (b) higher elastic moduli as the cohesion between the lamellae is higher. Since tungsten is a brittle material, the effect of the lower concentration of interlamellar cracks on these properties combined results in a lower thermal shock resistance [12]. On the contrary the best coating (No. 10) has been sprayed in nitrogen and at a longer spraying distance. It should have less good contact and be able to adjust itself more to the stresses induced by the thermal shocks. The spray atmosphere may also influence the temperature and velocity of the sprayed particles since the cooling of the plasma jet depends on the nature of atmosphere [13].

In the case of materials with low ductility such as tungsten, the density has many effects. Less porosity (with a given level of good contact between lamellae) results in a higher diffusivity and conductivity. This is a positive factor for heat extraction. However a coating containing

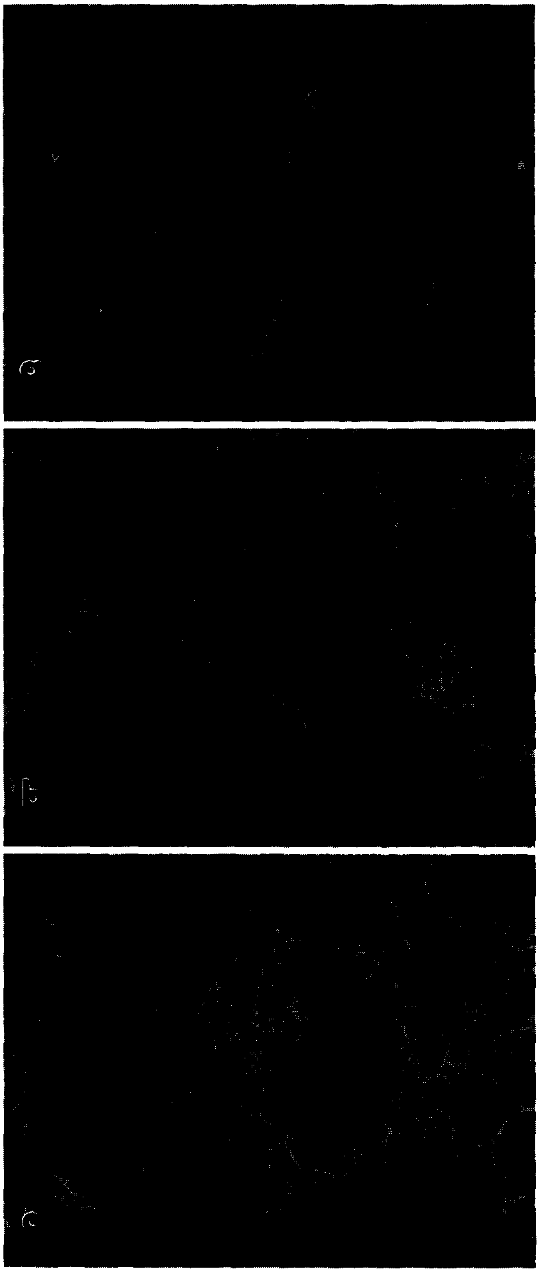

Fig. 3. SEM micrographs of the surface of LP (a: 22-2) and AP (b:7 and c: 3) after 40 shocks. Note the changes of scale.

Table 3
Summary of the observations on thick AP coatings

No.	Shocks for delamination unset	Edge delamination mode	Crack width (μm)
2α	40	sudden + small	12
β	20	sudden + small	15
4α	2–3	sudden	7
β	2–3	sudden	6
8α	15	progressive	6
β	20	progressive	8
9α	2–5	sudden	4
β	4–7	sudden	5
10α	> 100	—	12
β	37	progressive	8

less porosity is less resistant to thermal shocks as shown above. The porosity has also an effect on degassing, which is an issue in low pressure applications such as tokamak reactors. An optimum balance must therefore be reached between the thermal shock resistance and the degassing behavior of tungsten coatings.

4. Conclusion

This study of the thermal shock resistance of tungsten discusses the effects of several spraying parameters. Preliminary results at 60 MW/m^2 with 0.2 s shocks show that for 1 mm coatings, large cracks and, above all, delamination is observed after a variable number of shocks. For 100 μm coatings there are different modes of cracking but no delamination; in the denser coatings there are few cracks but they are wide, whereas in less dense coatings a network of numerous very thin cracks is found.

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