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Microstructure and tribological behavior of suspension plasma sprayed Al_2O_3 and Al_2O_3–YSZ composite coatings

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ABSTRACT

Several alumina and alumina–zirconia composite coatings were manufactured by suspension plasma spraying (SPS), implementing different operating conditions in order to achieve dense and cohesive structures. Temperatures and velocities of the in flight particles were measured with a commercial diagnostic system (Accuraspray®) at the spray distance as a function of the plasma operating parameters. Temperatures around 2000 °C and velocities as high as 450 m/s were detected. Hence, coatings with high amount of α-alumina phase were produced. The microstructure evolution according to the spray parameters was studied as well as the final tribological properties showing efficient wear resistance.

1. Introduction

Wear and corrosion are the major degradation modes that limit the service life of components. To protect pieces from wear, heat or corrosion, thermal spray and especially plasma spraying appears to be an efficient process [1]. Indeed, since the 1960s atmospheric plasma spraying (APS) has been widely used in industry and found applications in many fields like automotive, aeronautic, medical, and paper milling... [2,3]. Nowadays intensive researches are conducted on the elaboration of layers structured at the submicronic or even nanometric scale to reach better properties. Among the different possible routes to produce finely structured layers, suspension plasma spraying (SPS) appears as a possible one that can be easily implemented on current spray installation. Indeed, SPS is a recent process and an alternative to APS which allows producing finely structured and thinner layers due to the smaller size of the feedstock particles, ranging from a few tens of nanometers to a few micrometers, injected into plasma as a liquid suspension [4,5]. However, the set of parameters controlling the final coating architecture and properties are not yet fully mastered. Moreover, the functional properties of SPS layers are not intensively studied.

Consequently, this paper deals with the influence of some spray parameters on the SPS layer microstructure and try to highlight their important characteristics. The investigation of the architecture, the phase composition and the wear behavior are conducted and related to the processing parameters to obtain a better understanding of involved phenomena. Al_2O_3 and ZrO_2 have been used as demonstrative materials which can be potential candidates for several applications like wear barriers, thermal barrier coatings or solid oxide fuel cells.

2. Experimental methods

2.1. Suspension and injection

Suspensions of alumina and alumina–zirconia were made of angular single monocrystalline α-Al_2O_3 particles (P152 SB, Alcan, France) with a diameter (d_{50}) of 0.6 µm and ZrO_2 doped with 8 wt.% of yttria particles (HW 1429, Saint Gobain, France) with a d_{50} around 0.4 µm. The two powders were dispersed into distilled water with solid loads varying from 5 to 20 wt.%. The amount of anionic dispersant, PAA-NH_4 (ammonium salt of polyacrylic acid from Coatex, France) was adjusted to enhance the electrosteric dispersion of the particles following the previous works [6]. Prior to spraying, the suspensions were submitted to ultrasonic treatment for 5 min and then mechanically mixed to ensure a full dispersion of the particles as well as the suspension stability.

A pressurized gas delivery system feeds the slurry through an in-house two-fluid atomizing nozzle producing a spray of fine droplets to a conventional plasma torch. In this study, according to the injection parameters, the droplet size measured with the Spraytech (Malvern Instruments, France) varies from 10 to 40 µm with an average velocity at the nozzle exit around 35 m s^{-1} (measured by particle imaging velocimetry) as well as a feed rate of 0.83 g s^{-1}.

2.2. Spraying parameters and characterization apparatus

The spraying system consists of an atmospheric F4-VB plasma torch (Sultzer Metco AG, Switzerland), equipped with a 5, 6 or 8 mm

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internal diameter anode nozzle, and mounted on an ABB robot to ensure an accurate control of the overlapping coating kinematics. It was operated with a binary Ar–H_2 (45–15 NL min^{-1}) plasma forming gas mixture and with an arc current intensity of 500 A. Conventional measurements were performed to characterize the net power supplied to the plasma gas mixture and its specific enthalpy. Moreover, the average temperature and velocity reached by in flight particles at the spraying distance are measured with the use of the Accuraspray-g3 (Tecnar Automation, Canada).

The coatings were sprayed onto 314 L stainless steel coupons (50 mm in diameter and 5 mm in thickness) polished to an average surface roughness around 0.1 µm and degreased by immersion in an ethanol bath. The spray torch scan velocity and the scanning step are equal to 1 m s^{-1} and to 10 mm, respectively. The number of passes is 20, 40 and 80 respectively for a solid mass ratio of 20, 10 and 5 wt.%, to spray the same amount of solid material. At the end, the spray distance was varied from 40 to 25 mm.

The coating morphology was observed by SEM (Jeol JSM 5800 LV, France) and crystalline structure determined by XRD (Bruker AXS, D8Focus, France). The surface roughness and topography (of substrates and coatings) were depicted with a 3D profilometer (Altisurf 500, Altimet, France). The friction coefficient and the wear properties were assessed by a pin-on-disc test (S/N09-150, CSM Instrument, Switzerland) in the dry mode. The wear debris were continuously removed with an air jet located at the opposite of the contact point. A polished α-alumina (sintered) ball of 6 mm in diameter was applied with 2 N normal load at a sliding speed of 0.1 m s^{-1} for a sliding distance of 1500 m.

3. Results and discussion

Based on the T ratio hypothesis [7] that can depict and explain the final layer architecture as well as its structure evolution thanks to the evaluation of the number of poorly treated particles embedded into the layer compared with the quantity of well melted particles, the nozzle internal diameter (Ø), the spray distance (SD) and the solid mass ratio (SM) have been selected in the present study to assess their influence on the coating microstructure and wear properties.

3.1. Microstructure evolution versus operating parameters

First of all, in SPS process, to ensure good particle processing the suspension jet or spray has to be correctly injected into the plasma flow. Upon penetration, the suspension encounters two mechanisms, fragmentation and solvent vaporization. The fragmentation occurs when the drag force is higher than the surface tension forces and the final droplet size is reached when the two forces equalize. So, high plasma flow velocity may lead to the formation of smaller droplets. In turn, these small droplets may lead to in flight particles that are better melted and accelerated until their impact onto the substrate showing finely structured layers with a high molten state of the particles.

Moreover the increase of the plasma flow velocity is able to wipe out more poorly treated particles at the boundary layer perpendicular to the plasma torch axis, which are responsible for the stacking defects. Finally, it also entails the decrease of the boundary layer thickness leading to a better energetic transfer from the plasma to the substrate, and so a better cooling and flattening of the lamellae.

Thus, the plasma flow velocity was increased by decreasing the anode nozzle internal diameter [8]. At SD of 30 mm, the impacting particle velocity measurements are equal to 350, 470, and 500 m s^{-1} for an internal nozzle diameter of 8, 6 and 5 mm, respectively. Furthermore, the observation of the alumina coating cross sections shown in Fig. 1 lets appear a denser and more cohesive structure with the decrease of the nozzle internal diameter. The increase of the plasma velocity through the decrease of the torch nozzle diameter has a major effect on the particle velocity and thus on the coating microstructure.

A better energetic transfer from the plasma to the particles can also be achieved by the injection of less loaded suspensions. Thus, for the same plasma flow, the available energy to process the particles is higher and better melting and acceleration are expected. Fig. 2 shows different cross section views of SPS coatings produced with the same operating conditions except the solid loads in the suspensions. The number of passes was adjusted to spray the same amount of solid material. As the solid mass ratio decreases from 20 wt.% down to 10 wt.% and 5 wt.%, the thickness of the layer decreases from 28 µm to 20 µm and finally 8 µm respectively. In the meantime it is clearly evidenced that the morphology of the coating is changing from a very porous one to a rather dense structure. The decrease of the deposition efficiency could be a part of the thickness reduction explanation but as porosity evolves the stacking ability changes and could favor thin and dense coating. Indeed, by decreasing the amount of solid particles it can be assumed that the agglomeration mechanism within the in flight liquid drops is lower leading to the formation of thinner molten particles impacting onto the substrate which promotes the manufacture of more finely structured layer. In contrast, high solid mass

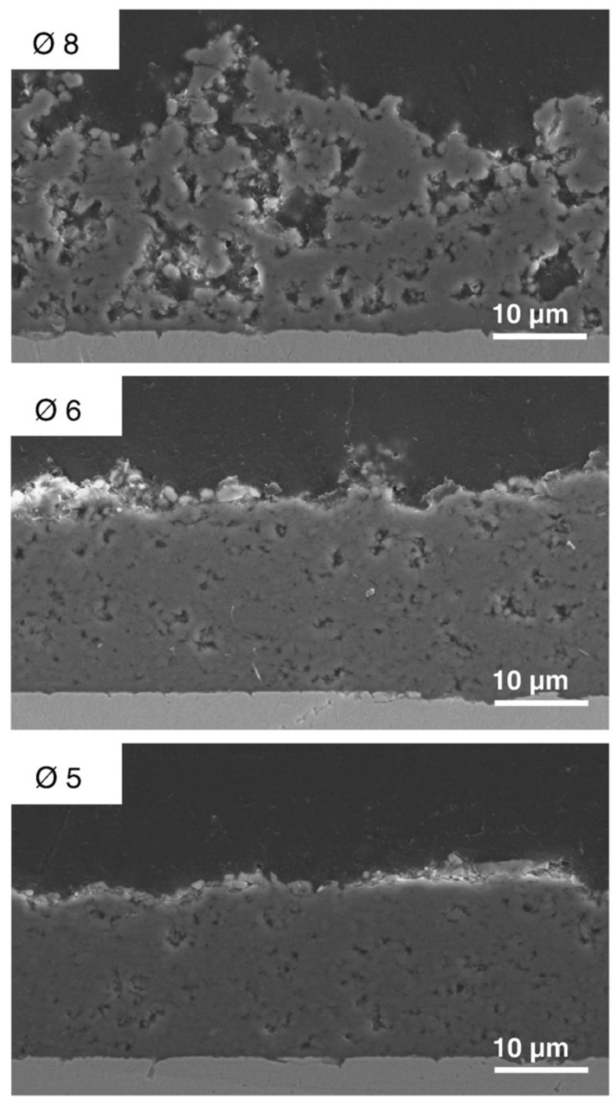

Fig. 1. SPS coating microstructure evolution function of the internal anode nozzle diameter, Ar–H_2 45–15 NL min^{-1}, I_{arc} = 500 A, $\overline{h}$ = 14.5 MJ kg^{-1}, SD = 30 mm, SM = 10%.