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Suspension plasma sprayed TiO_2 coatings using different injectors and their photocatalytic properties

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ABSTRACT

Stable water- and water/ethanol suspensions of TiO_2 were plasma sprayed on stainless steel substrates. The suspensions were injected using two different systems: external, using an atomizing injector, and internal, performed with a continuous-stream injector inside the plasma torch anode. In order to find the optimal spray parameters, seven experimental runs were performed and the resulted deposits were mainly characterized by means of scanning electron microscopy and X-ray diffraction analysis. The microstructural examinations revealed that the coatings obtained by internal injection of the suspension show two principal characteristics: *dense zones* with well molten big lamellae and *porous zones* containing fine nanometric and submicrometric grains. When applying the internal continuous-stream injector, the amount of anatase varied from about 1.6 vol.% to 8.8 vol.%. The amount was with 9.4 vol.% to 15.4 vol.% considerably lower when an external atomizing injector was used. The photocatalytic activity of selected coatings was determined by measuring the degradation of an aqueous solution of dye methylene blue. The photon efficiency of titania deposits, which is a measure of the photocatalytic performance, varied from 0.022% up to 0.032% and did not seem to depend on the amount of anatase phase.

1. Introduction

Due to their interesting physical, chemical and electrical properties, titanium oxide (TiO_2) films and coatings have been intensively studied in the past years. TiO_2 coatings found large applications in new fields such as hydrogen and oxygen sensors [1,2], self-cleaning and photocatalytic surfaces for the degradation of organic pollutants [3–5], destruction of some types of bacteria [6–8] and electron emitters for light emitting devices [9,10]. These coatings are generally obtained using thin film deposition methods such as physical and chemical vapour deposition, sol–gel and, more recently, electrophoretic deposition [11]. Thermal spraying is largely used to obtain titanium oxides coatings designed for mechanical, electrical, solid-lubrication, biomedical applications and so on. A review on the characteristics of TiO_2 thermally sprayed coatings was made by Berger [12]. In the last years, thermal spraying has gained more and more interest for the preparation of photocatalytic active titania surfaces. Conventional thermal spraying methods involve spraying coarse powder particles which produce relatively thick coatings (generally higher than 100 µm). The new spray method with liquid feedstock, the so-called suspension spraying technique makes it possible to obtain coatings with thickness from a few micrometers to hundred of micrometers [13–19]. In this technique, a suspension of fine nanometric and submicrometric powder particles can be injected in a plasma (suspension plasma spraying method, SPS) or in an HVOF flame (Suspension HVOF method, S-HVOF/SHVSFS). In the present work water and water/ethanol suspensions of fine TiO_2 particles were internally injected in the plasma torch through a continuous-stream nozzle injector in order to obtain photocatalytic active titania coatings. The characteristics of the coatings (microstructure and phase compositions) were compared to those of the coatings obtained by external injection of the suspension into the plasma jet as described in [20,21]. The photocatalytic activities of the latter were tested by the degradation of an aqueous solution of dye methylene blue and the photon efficiency was determined.

2. Experimental

2.1. Suspension preparation and injection method

A rutile, TiO_2 *Tioxide R-TC90* powder (*Huntsman Tioxide*, England) with a mean diameter (volume-surface mean defined by Masters [22]) of about $d_{VS} = 0.33$ µm was used for the suspension formulation. The morphology and chemical properties of the powder are carefully described in [16]. Stable TiO_2 suspensions were prepared by dispersion of 10 wt.% and 20 wt.% of powder in water and water/ethanol (H_2O: $C_2H_5OH = 1:1$). The suspensions were supplied by a pneumatic system

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(reservoir) as described elsewhere [20]. The suspension feed rate varied with the static pressure in the container, ranging from 17 g/min at 0.040 MPa up to 27 g/min at 0.058 MPa. The radial injection of the suspension feedstock was made via two modes: (i) external injection mode (Fig. 1a) using an atomizing injector, where the suspension is firstly atomized with help of an inert gas before entering the plasma [18] and (ii) internal injection mode (Fig. 1b) using a continuous-stream injector (with internal diameter of 0.5 mm) inside the anode-nozzle of the plasma torch.

2.2. Plasma spray parameters

Plasma spraying was performed using an *SG-100* (*Praxair S.T.*, Indianapolis, In, USA) DC single cathode torch mounted on a 5-axis *IRB-6* robot of *ABB* (Zürich, Switzerland) using an Ar–H_2 (45–5 slpm) plasma gas mixture with electric power varied from 30 kW to 40 kW. Different process parameters (injection mode, suspension feed rate, spray distance, plasma power) were tested with an aim to obtain well adhering coatings including molten particles. More details about the suspension spraying using the atomization mode can be found in the previous work [18]. The deposits were sprayed with a bypass of 3 mm and after each torch scan the deposition was stopped and the coatings were cooled down to the temperature of 30–40 °C. The suspension spraying was performed in order to obtain coatings with thickness up to 100 μm. Stainless steel plates (15 × 15 × 3 mm) previously cleaned with ethanol and sand blasted using corundum grit under a pressure of 0.4 MPa were used as substrates. The operational process parameters realized with nozzle injector for suspension are summarized in Table 1.

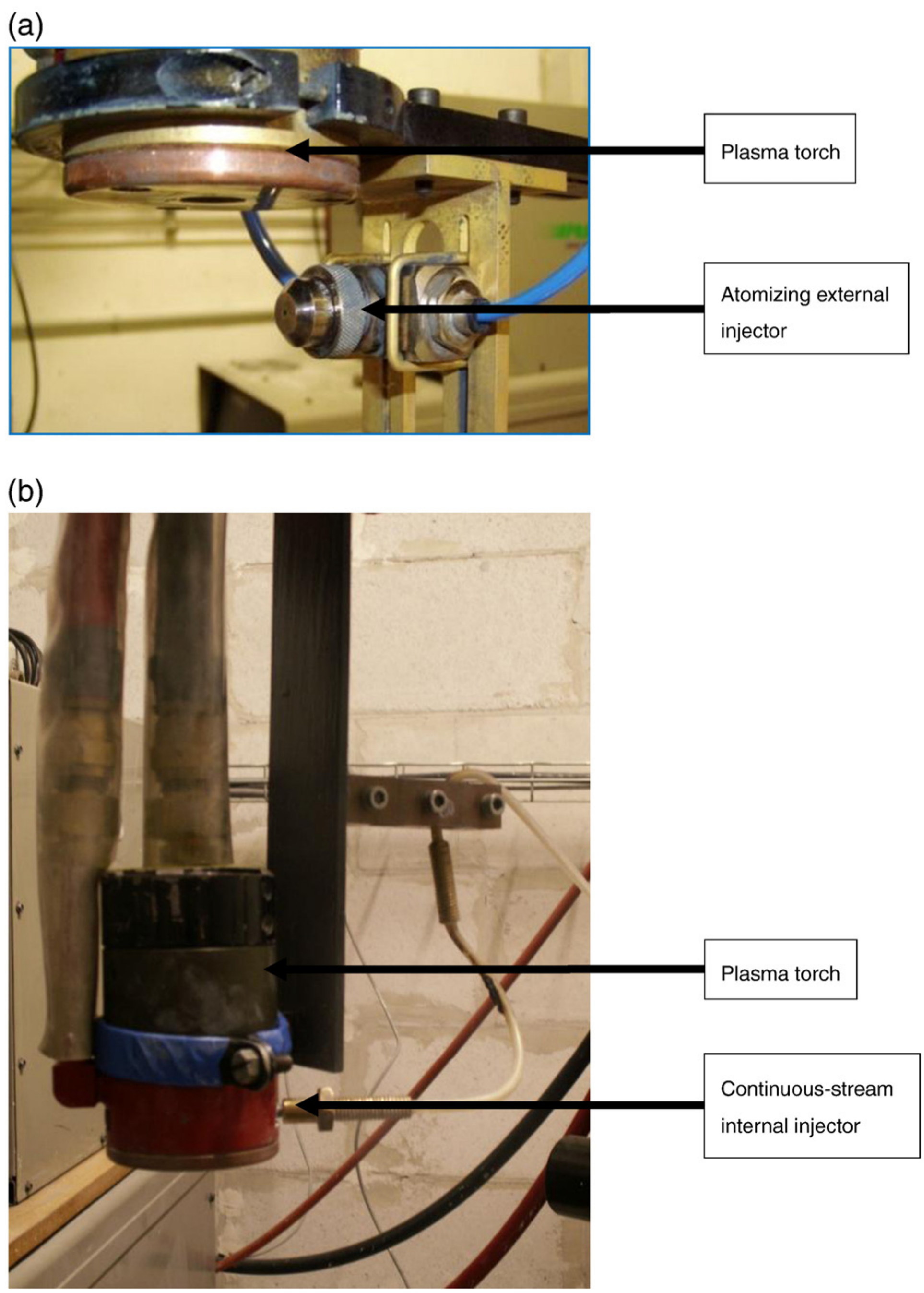

Fig. 1. Set-ups of the suspension injectors: (a) external injection mode – atomizing injector; and (b) internal injection mode – continuous jet nozzle injector installed inside the plasma torch.