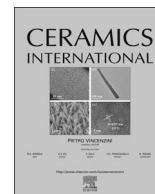




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# Dense yttria-stabilized zirconia coatings fabricated by plasma spray-physical vapor deposition

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## ABSTRACT

Plasma spray-physical vapor deposition (PS-PVD) is a novel technique which can offer new opportunities to obtain advanced microstructures. In this study, dense yttria-stabilized zirconia (YSZ) coatings were deposited using PS-PVD with proper parameters. The dependencies of the microstructure of the as-sprayed coatings on axial spray distance and radial position were discussed. Four typical coatings were selected for the current work. The corresponding microstructures, phase compositions and mechanical properties were studied in detail. These as-sprayed coatings had dense lamellar or lamellar/columnar hybrid microstructures, the lowest porosity was 0.44%. Both monoclinic and tetragonal zirconia were detected in the as-sprayed coatings. The distribution of yttrium was not homogeneous, especially in the coatings deposited at shorter spray distance. Furthermore, oxygen got lost partly during the spraying process with decreasing spray distance. The hardness ( $H$ ) and Young's modulus ( $E$ ) of the coatings changed simultaneously with the different microstructures. The maximum values of  $H$  and  $E$  were  $16.6 \pm 0.6$  and  $234.3 \pm 10.1$  GPa respectively, which were found in the densest coatings. The dense PS-PVD ceramic coatings might be applicable in anti-corrosion fields (such as alkali corrosion, marine corrosion and hot corrosion).

## 1. Introduction

Yttria-stabilized zirconia (YSZ) coatings have been widely applied due to their excellent mechanical, chemical, thermal properties and high ionic conductivity [1–3]. As reported, YSZ coatings have been employed as anti-corrosion coatings in acidic (0.5 M H<sub>2</sub>SO<sub>4</sub>), neutral (3.5 wt% NaCl), basic (0.1 M KOH) solutions and artificial seawater [4,5]. Meanwhile, YSZ coatings are also found on gas turbines, used as thermal barrier coatings (TBCs). As for their deposition process, various methods have been used, such as air plasma spraying (APS), electron beam-physical vapor deposition (EB-PVD), sol-gel, aerosol deposition [6–9]. APS technology is extensively used in industrial scale to deposit YSZ coatings because of high deposition rate, economical efficiency and ability to produce thick coatings with high adhesive strength. However, a high porosity (about 5–8%) in ceramic coatings using conventional APS is almost inevitable [10]. High porosity in YSZ coatings is beneficial to thermal insulation, for it usually reduces the effective thermal conductivity of coatings, whereas, it is deleterious for anti-corrosion purpose [5,11,12]. On the one hand, higher porosity means more corrosive species penetrating into the coatings, thus denser ceramic coatings exhibit higher corrosion resistance [13,14]. On the other hand, YSZ coatings might be covered by molten salt when

served as TBCs in the hot section of an engine. The penetration of these contaminants through the porous and microcracked coatings may attack the underlying superalloy by hot corrosion mechanisms [15–17]. For conventional APS YSZ coatings, sealing and laser glazing have been used to improve the hot corrosion resistance by forming the dense layer [15–17]. Vapor phase deposition processes (such as chemical vapor deposition and physical vapor deposition) can produce dense ceramic coatings, but their applications are seriously restricted by the high investment cost and low deposition rate [18].

In recent years, plasma spray-physical vapor deposition (PS-PVD) has emerged as a new promising technology to produce advanced microstructures for the ceramic coatings and thus to meet the increasing demands on modern functional coatings [19,20]. PS-PVD combines the advantages of plasma spraying and vapor deposition. Owing to the low pressure during the depositing process, the plasma jet can expand to 2 m long and 200–400 mm in diameter [21]. With a high plasma torch, which allows a high total gas flow of up to 200 SLPM and high power levels up to 180 kW (3000 A), high enthalpy plasma stream can be obtained [22]. As a result, the coatings with high density and large size can be deposited by PS-PVD at an unprecedented speed. Besides, the feedstock can be partially or almost completely evaporated. Some previous literatures have reported the microstructures and properties

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of the YSZ coatings fabricated by PS-PVD [23–25]. However, the reports mainly focus on columnar structures. Li et al. and Mauer et al. [26,27] briefly introduced the dense PS-PVD YSZ coatings deposited at shorter spray distance. Zhu et al. [18] prepared dense  $\text{Al}_2\text{O}_3$  and YSZ ceramic coatings with an average thickness of 30–40  $\mu\text{m}$  by very low pressure plasma spray (VLPPS) process. The coatings were expected to contribute to the applications of electrical insulation, SOFC, TBCs, membrane, wear-resistance, etc.

In the current work, dense YSZ coatings were successfully prepared by PS-PVD process. To exploit the potential of PS-PVD technology, the dependency on process conditions should be well understood. Apart from the spray distance, the radial location of the samples has a significant influence on the coating structure as well. Thus, coatings deposited with different spray distances and radial distances were investigated. At decreasing spray distance and edger regions, dense coatings can be obtained. Since porosity is one of the main microstructural features affecting the properties of the coatings, porosity details of the PS-PVD YSZ coatings were studied specially. Hardness ( $H$ ) and Young's modulus ( $E$ ) of the coatings were measured to evaluate the mechanical properties of coatings and to gain a better understanding of the relationship between coating microstructure and its mechanical property. Based on the results and literatures, the formation mechanism of dense coatings was discussed. The dense YSZ coatings in this study can probably work in the environments where both wear and corrosion resistances are required, especially at an elevated temperature. Furthermore, the process might be able to expand to get other ceramic coatings with dense microstructures, e.g.  $\text{Al}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ . Meanwhile, the dense YSZ obtained in this study might be applicable in TBC systems as the top layer on the columnar layer. Due to the reduced specific surface area of the dense YSZ layer, the corrosion reaction between the molten salts and the YSZ in the bilayered coatings is likely to be lower than that of only columnar structured YSZ coatings. However, further researches for practical use must be performed before the specific application is determined. This is only at an early stage of the feasibility study.

## 2. Experimental procedure

### 2.1. Coating preparation

The coatings were fabricated using an Oerlikon Metco (Switzerland) LPPS hybrid system. The polished rectangle graphite blocks were used as the substrates. The spray procedure is illustrated in Fig. 1. The spray distance was defined as the distance between the nozzle exit of spray gun and the substrate surface. Spray distances ranging from 500 mm to 800 mm at an interval of 100 mm were applied. To determine the radial location precisely, 19 graphite blocks with dimension size of L:50 mm×W:10 mm×T:9 mm were used in each deposition process. The feedstock material was an agglomerated YSZ powders supplied by Oerlikon Metco (Metco 6700). Metco 6700 has a spheroidal morphology with a particle size distribution of  $-30+1\ \mu\text{m}$ , and the median particle diameter ( $D_{50}$ ) is 10  $\mu\text{m}$ . The deposition parameters are summarized in Table 1. Before coating deposition, the chamber pressure was lowered down to about 1.5 mbar and the substrates were preheated to above 1000  $^{\circ}\text{C}$  by the plasma jet. The obtained samples were labelled as (1–19)–(500–800), where the former is the number of the substrate (as shown in Fig. 1) and the latter represents the spray distance (mm). No. 11 samples were located at the jet axis. The positions of spray gun and samples were kept constant in all cases.

### 2.2. Coating characterization

The morphologies of the YSZ coatings were characterized by a scanning electron microscope (SEM, TM3000, Hitachi, Japan) with affiliated 3D-view device. All of the SEM images were taken in the secondary electron (SE) mode. An energy-dispersive X-ray spectro-

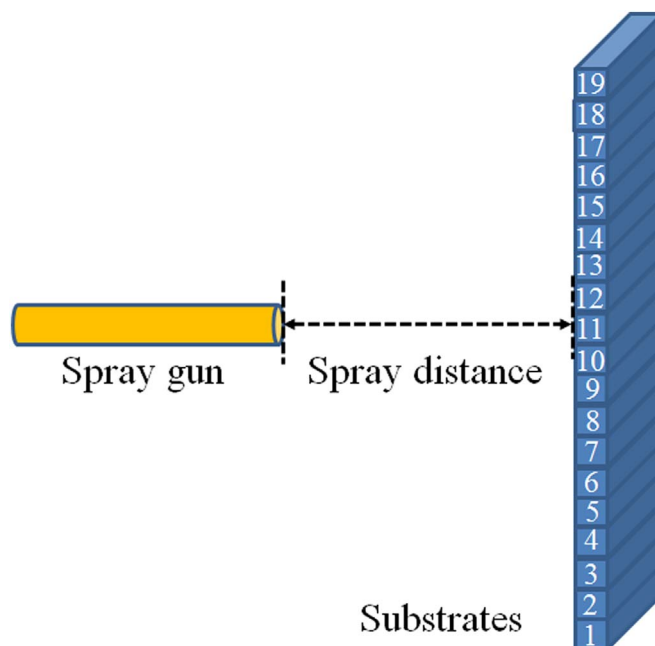


Fig. 1. Schematic diagram of the PS-PVD procedure.

Table 1  
PS-PVD operating parameters.

Parameters	Value	Unit
Current	2600	A
Net power	65	kW
Primary plasma gas (Ar)	35	slpm
Secondary plasma gas (He)	60	slpm
Carrier gas (Ar)	8	slpm
Powder feed rate	5	g/min
Spray distance	500–800	mm
Spray duration	50–125	s

meter (EDS, Swift ED 3000) equipped on the SEM was employed to evaluate the element compositions of the coatings. Porosities of the coatings were estimated by an image analysis (IA) method using Image-Pro Plus software (version 6.0). For each coating sample, 5 micrographs (with the amplification  $\times 5000$ ) at random locations on the cross section were analyzed. The roughness of the as-sprayed coatings was measured by a roughometer (T8000, R120-400, Jenoptik Industry Metrology GmbH, Germany). The phase compositions of the powders and coatings were identified by X-ray diffraction (XRD, D/max 2550 V, Rigaku Tokyo, Japan). Nanoindentation experiments were performed for determining the hardness and Young's modulus of the coatings [28]. A G-200 nanoindenter (Agilent Technologies, Oak, Ridge, USA) equipped with a Berkovich-type indenter was used and the instrument was operated in the continuous stiffness mode (CSM). The maximum indentation depth was 5000 nm, the values at a penetration depth of about 500 nm were applied to represent the results. 10 separate indentations were made on the cross section of each investigated sample. A Poisson's ratio of 0.25 was used to calculate the Young's modulus.

## 3. Results

### 3.1. Microstructure

It is well known that the microstructure has a significant influence on the corrosion resistance of the coatings. Structural defects can provide paths for corrosive species, leading to a deleterious effect on the coatings performed in aggressive environments. Therefore this

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