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# 7YSZ coating prepared by PS-PVD based on heterogeneous nucleation

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## KEYWORDS

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**Abstract** Plasma spray-physical vapor deposition (PS-PVD) as a novel coating process based on low-pressure plasma spray (LPPS) has been significantly used for thermal barrier coatings (TBCs). A coating can be deposited from liquid splats, nano-sized clusters, and the vapor phase forming different structured coatings, which shows obvious advantages in contrast to conventional technologies like atmospheric plasma spray (APS) and electron beam-physical vapor deposition (EB-PVD). In addition, it can be used to produce thin, dense, and porous ceramic coatings for special applications because of its special characteristics, such as high power, very low pressure, etc. These provide new opportunities to obtain different advanced microstructures, thus to meet the growing requirements of modern functional coatings. In this work, focusing on exploiting the potential of gas-phase deposition from PS-PVD, a series of 7YSZ coating experiments with various process conditions was performed in order to better understand the deposition process in PS-PVD, where coatings were deposited on different substrates including graphite and zirconia. Meanwhile, various substrate temperatures were investigated for the same substrate. As a result, a deposition mechanism of heterogeneous nucleation has been presented showing that surface energy is an important influencing factor for coating structures. Besides, undercooling of the interface between substrate and vapor phase plays an important role in coating structures.

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## 1. Introduction

The plasma spray-physical vapor deposition (PS-PVD) process has been developed with the aim of depositing different structured functional coatings (such as thin, gas tight, columnar coatings, etc.) with large area coverage by plasma spray.<sup>1-3</sup>

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The PS-PVD is developed based on low-pressure plasma spray (LPPS), where electrical current up to 3000 A, plasma gas flow up to 200 L/min, and an input power level of 180 kW could be achieved. With operation pressure decreasing, the plasma plume expands to a length of more than 2200 mm and a diameter of 400 mm. Using appropriate parameters, it is possible to evaporate powder feedstock materials, resulting in variant microstructures and non-line-of-sight deposition.<sup>4,5</sup> Li et al.<sup>6</sup> reported that using an advanced PS-PVD process, the nano-hardness and micro-hardness of prepared dense coatings were markedly higher than those of conventional YSZ coatings (i.e., coatings fabricated by atmospheric plasma spray or electron beam-physical vapor deposition), even comparable with those of sintered YSZ polycrystal. Goral et al.<sup>7</sup> presented that columnar YSZ coatings were deposited from evaporating powders during the process of PS-PVD. The microstructures of coatings were affected by the feed rate, chamber pressure, sample rotation rate, and plasma gas ratio (Ar, He, and N<sub>2</sub>). Hospach et al.<sup>8</sup> produced columnar-structured YSZ coatings with a diameter between 20 and 750 μm through PS-PVD. Thinner and thicker coatings seem to be possible. The geometry and arrangement of a sample and the sample holder have a big influence on the coating quality.

Despite much investigation about PS-PVD have been done in the past few years, however, the basic process technology, such as heating of powder particles to spray-deposited molten, semi-molten droplets or vapor gas phase onto substrate surface, has remained essentially the same.<sup>9,10</sup> There are still a lot of areas which have not been investigated thoroughly. These areas consist of particle-plasma interactions in the rarefied plasma, particle vaporization and its affect on plasma properties, and deposition mechanisms associated with different microstructures. Now, any further understanding in each of these areas will enable the spray community to more easily apply the PS-PVD technology to meet emerging coating challenges.<sup>11,12</sup>

To exploit the potential such as gas-phase deposition by PS-PVD, the deposition mechanisms and their dependency on process conditions must be better understood. The PS-PVD process can be summarized as three steps<sup>9</sup>: (A) feedstock processing in plasma torch; (B) plasma jet formation and materials transport; and (C) deposition and coating growth. The third step mainly includes heterogeneous and homogeneous nucleation depending on the spray distance.<sup>13</sup> When the spray distance is set at the middle area in the axial direction of the plasma flame, coating deposition primarily relies on heterogeneous nucleation on the substrate surface. In this work, different structured coatings based on heterogeneous nucleation have been obtained, and these principles are summarized in this investigation.

## 2. Experimental procedure

The experimental set-up is based on an Oerlikon-Metco Multicoat™ PS-PVD system together with an O3CP plasma torch mounted on a robot manipulator of ABB inside a  $\varnothing 2.5 \text{ m} \times 4.5 \text{ m}$  vacuum chamber. The PS-PVD system is obtained through a comprehensive reconstruction of an existing conventional LPPS system. In particular, the system is equipped with an additional vacuum pumping unit, a large vacuum blower to provide sufficient pumping capacity at low

pressures and enhanced cooling capacity, additional power sources, a new torch transfer arc system, and new operational control units. In terms of the powder feeding system, two powder injectors are located in the cylindrical section of the O3CP nozzle (diameter = 12.5 mm) close to the divergent part.

Feedstock agglomerated 7YSZ powders (Metco 6700, Oerlikon-Metco) were used and their grain sizes ranged from 5 to 22 μm. 7YSZ coatings were deposited on graphite, sintered zirconia, and nickel-based superalloy K417 substrates (size  $\varnothing 25.4 \text{ mm} \times 5 \text{ mm}$  and surface roughness  $< 2 \mu\text{m}$ ) at a spray distance of 950 mm, where the Ar-He hybrid plasma was operated at a 67 kW net power of O3CP under an operation pressure of 150 Pa. Meanwhile, the substrate pre-heating temperatures were controlled at 850 °C and room temperature prior to deposition of 7YSZ coating, respectively. During the pre-heating or deposition process, the substrate remained still, while the plasma gun moved at a speed of 1000 mm/s. The detailed spray parameters are shown in Table 1.

The microstructures of 7YSZ coatings were characterized by field emission-scanning electron microscopy (FE-SEM, Nova-Nano430, FEI) and transmission electron microscopy (TEM, JEM2100F, JEOL). Additionally, before TEM characterization, test samples were prepared by focused ion beam (FIB, 450S, FEI) milling.

## 3. Results and discussion

### 3.1. Variation of coating microstructure

#### 3.1.1. Effect of substrate materials

Taking graphite as a substrate without pre-heating by plasma flame (namely, the substrate temperature is controlled at room temperature), 7YSZ coatings prepared by PS-PVD show typical columnar microstructure, as seen in Fig. 1(a). The interface between the coating and the substrate has good bonding without crack. Among columns, there exist different sizes of gaps. Between columns, many small particles were formed, which were resulted from condensation of the vapor phase<sup>14,15</sup>, as shown in the magnified Fig. 1(b). When the graphite substrate was replaced by sintered zirconia, similar columnar 7YSZ coating was generated with the same parameters at room temperature, as presented in Fig. 2(a) and (b). According to the result of comparison, it can be known that the horizontal width of a single column is larger than that of a column deposited on graphite. Moreover, the deposition rate of 7YSZ on graphite is higher than that on sintered zirconia due to different thermal conductivities between graphite (129 W/(m·K)) and zirconia (2.2 W/(m·K)). During the deposition process, the temperature gradient on graphite is larger than that on zirconia, which results in a higher growth driving force on graphite. Thus, graphite has a higher deposition rate. Due to similar properties, the interface between 7YSZ coating and sintered zirconia has a better bonding. Between columns, there is no apparent vertical gap, and no small particle as well appears in the gaps. Besides, the columns are made of fine grains and denser than those deposited on graphite.

#### 3.1.2. Effect of substrate temperature

With a net power of 60 kW, the inner O3CP gun provided high plasma energy density, and the electron excitation temperature is more than 10,000 °C, so that most of the 7YSZ powders can

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