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Microstructure formed by suspension plasma spraying: From YSZ splat to coating

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ABSTRACT

In the current work, by means of suspension plasma spraying (SPS), yttria stabilized zirconia splats and coatings were deposited onto mirror-polished and rough substrates, respectively. The splats were characterized by scanning and transmission electron microscopes (TEM). The influence of substrate temperature on splat morphology was analyzed via comparing the splats deposited onto substrates held at different temperatures, namely room temperature (RT), 300 °C, 500 °C, 700 °C and 900 °C. As the substrates were heated from RT to 300 °C, the feature of splats changed rapidly. However, when the substrate temperature further increased, the variation of morphology was found less conspicuous. Integrated splats were peeled off from the substrate using a "plating and etching" approach for TEM examination. The observation on morphological and crystallographic features of splats revealed that the cooling rate during impacting and spreading of SPS splats was lower than that of splats deposited by conventional atmospheric plasma spraying. Based on the characteristics of splats, a modified mechanism consisting changing of droplets trajectory and shadowing effect was proposed to explain the formation of the microstructure of as-sprayed SPS coating. In addition, the coatings were also examined by X-ray diffraction and nano-indenter to determine the phase composition and microhardness, respectively.

1. Introduction

Atmospheric plasma spraying (APS) is a well-established process to fabricate yttria stabilized zirconia (YSZ) thermal barrier coatings (TBCs) owing to its high depositing rate, low cost and versatility, etc. [1]. With splats piling up, the as-sprayed coating exhibits a major microstructural feature of inter-splat cracks and spheroidal/ellipsoidal pores. In spite of its lower thermal conductivity, such architecture inevitably leads to unfavorable durability and reliability [2]. Therefore, fine YSZ coatings with nano-/submicro-sized microstructure have attracted increasing attention in the community.

Currently, three different routes based on APS have been successfully carried out to prepare advanced fine YSZ coatings, namely:

I) spraying agglomerated nano-sized particles with conventional APS equipment;
II) spraying solution precursors with liquid carrier (known as solution precursor plasma spraying, SPPS);
III) spraying suspension with liquid carrier (known as suspension plasma spraying, SPS);

Over the last decade, a number of studies and overviews [3–9] have been published, focusing on the influences of feedstock/suspension/solution characteristic and spraying parameters on the coating microstructure. In order to further improve the coating performance, collective efforts of the community have been continuously made to have a more comprehensive understanding of this correlation.

Earlier, another paper of our group reported the differences in thermal shock behavior and failure mechanism between thick TBCs prepared by SPS and conventional APS [10]. The aim of our current work is to compare the different manners in splat formation and detailed coating microstructure. Since the flattening and solidification behaviors of APS splats have been extensively reported [11–14], this paper is primarily devoted to splats and coatings fabricated by SPS.

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2. Material and methods

2.1. Experimental procedures

Nickel-based superalloy GH3128 (Ni-20Cr-8Mo-8W-0.5Al-0.5Ti, wt%) substrates with dimensions of Φ10 mm×8 mm were used to accommodate individual splats. They were mirror-polished via a general multi-step process and ultrasonically cleaned in ethanol before spraying. In order to obtain distinguishable splats, a stainless steel shield with a Φ2 mm hole was placed in front of the substrate to reduce the amount of deposited splats.

The substrate temperature was controlled by an inductive heater along with a pyrometer and a digital controller. The spraying began once the substrates were heated to preset temperature by this home-made system. The temperature was stabilized within a narrow range during the whole process.

As for complete coatings, the GH3128 substrates with dimensions of 25 mm×25 mm×2.7 mm were sand-blasted. Bond coatings (BC) were fabricated by a high velocity oxy-fuel (HVOF) gun DJ2600 (Sulzer Metco AG, Switzerland) using commercial AMDRY 995 (Co-32Ni-21Cr-8Al-0.5Y, wt%, Sulzer Metco, USA).

The feedstock (see Fig. 1) for both ceramic splats and top coatings was aqueous suspension of 8YSZ (ZrO_2-8Y_2O_3, wt%, Jiangsu LIDA Hi-tech Special Material Co., Ltd., China). The median diameter of ceramic particles was 60 nm and the proportion of solid phase was about 20 wt %. The Zeta-potential of this commercial suspension at 25 °C was 36.0 ± 3.0 mV. The suspension was properly dispersed by ultrasonic processing before loaded into the feeder.

Splats and coatings were deposited by a plasma gun (Axial III 600, Northwest Mettech Corp., Canada) equipped with NanoFeed 350 suspension feeder (Northwest Mettech Corp., Canada). In this system, the atomized suspension was axially injected into the core of the plasma. Recipes for YSZ splats and top coatings could be found in Table 1, while minor details such as spraying parameters for metallic bond coatings were found in Ref. [10]. Reduced feeding rate of suspension was applied for splats deposition because interfering SPS splats were not expected.

2.2. Characterization

Field emission scanning electron microscope (FESEM) JSM-6700F (JEOL, Japan) was employed to observe the SPS splats. Over 2500 splats were marked and their diameters were measured with the help of software Image-Pro Plus (version 6.0.0.260). The observed areas were randomly chosen and the magnifications were kept the same (×10,000). Quantitative results such as diameter distribution, median diameter and splat density (defined as the amount of splats per unit area, namely μm^{-2} in the current work) were acquired from diameter statistics mentioned above.

Table 1
Spray parameters for YSZ splat and top coating.

Parameter (unit)	Splats	Top coating
Plasma gas flow (slpm)	Ar: 170–176. N_2: 48–53. H_2: 20–25	
Carrier gas flow (slpm)	N_2: 15	
Standoff distance (mm)	60–90	
Nozzle diameter (mm)	9.5 (3/8 in.)	
Suspension feed rate (ml/min)	10	40
Power (kW)	~110	

Transmission electron microscopy (TEM) images of splats and cross sections of coatings were acquired on JEM-2100F (JEOL, Japan). TEM specimens of coatings were wedge-shaped. They were obtained from the coating surface by focused ion beam (FIB) sputtering. Meanwhile, TEM specimens of ceramic splats were peeled off by an etching procedure, as demonstrated in Fig. 2: First, the sprayed substrates were coated with carbon. The substrates were then scarified to form a pattern of squares with a side of several millimeters. The scratches would act as channel for acid. After that, the substrates were immersed in alcohol-nitric acid solution. The splats inserted into the carbon film would leave the substrate after hours of immersion because the metallic material bonded with the carbon film and ceramic splats were fretted. The concentration of nitric solution employed in this work was 20 vol%. Then carbon films embed with splats were brought up using a copper grid. At last, the grid was cleaned for TEM observation.

The microstructure of as-sprayed coatings was examined by a scanning electron microscope (SEM) TM3000 (HITACHI, Japan). The phase composition was identified by X-ray diffraction (XRD) employing D/Max 2550V (Rigaku, Japan) with nickel-filtered Cu Kα radiation (λ=0.154 nm) at a scan rate of 0.5°/min. Continuous stiffness measurements were carried out using a nano-indenter G200 (Agilent Technologies, USA) on the polished cross section of as-sprayed coatings. The strain rate was 0.05 s^{-1} and the maximum indentation depth was 5000 nm. The mean value of 10 results obtained at the depth of 500 nm was viewed as the microhardness.

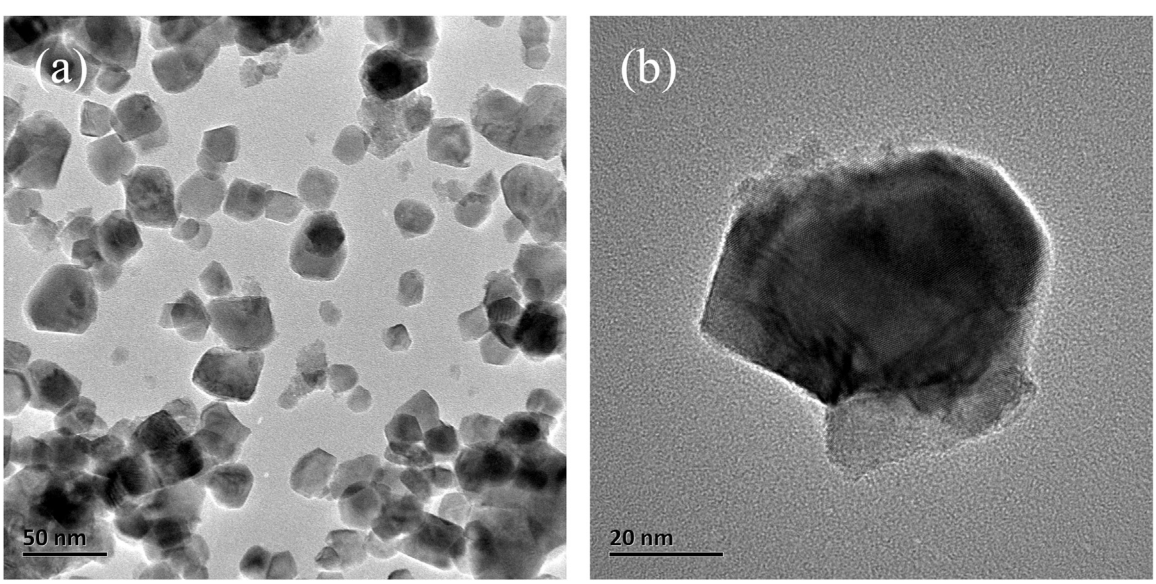

Fig. 1. TEM images of the starting suspension.