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Microstructure and mechanical property of dense yttria-stabilized zirconia coating fabricated by an axial bi-cathode plasma torch under very low pressure

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Abstract

Very low-pressure plasma spray is a promising technique for preparing dense metal/ceramic coatings due to the high-speed plasma jet. However, at the same time, the low-pressure environment weakens the ability of the plasma jet to heat sprayed materials. In order to improve this shortcoming, a novel bi-cathode plasma torch was firstly designed in this study, which could enhance the plasma power from 40 kW to 81 kW and realize the axial powder feeding. By using this torch under 6 mbar, a dense and uniform yttria-stabilized zirconia coating with a porosity of 2.7% was fabricated, which had a hybrid microstructure that deposited with the form of unmelted particles, melted droplets, and vapor clusters. Meanwhile, 40% of the zirconia phase changed from the monoclinic type to the tetragonal type, indicating the yttria-stabilized zirconia powder was melted well. The microhardness and elastic modulus of the coating was high compared with other plasma spraying process that using similar power, which was 11.7 ± 0.6 GPa and 167.8 ± 4 GPa, respectively. The high density and mechanical property of the YSZ coating demonstrated that using the bi-cathode plasma torch at low pressure has great potential for the preparation of SOFC electrolyte.

Keywords: dense yttria-stabilized zirconia coating; axial plasma torch; very low-pressure plasma spray; microstructure; mechanical property

1. Introduction

Yttria-stabilized zirconia (YSZ) coating has been widely used in industry due to its excellent properties such as low thermal conductivity, high ionic conductivity, and good mechanical strength [1-6]. In the field of energy, YSZ is the most commonly used electrolyte material for solid oxide fuel cells (SOFCs). It plays two important roles during SOFC operation, one is to conduct oxygen ions, and the other is to isolate fuel and oxidant. Both require the density of YSZ coating to be very high, usually greater than 95%. Many deposition methods can be used to produce YSZ coating, such as plasma spraying (PS), physical or chemical vapor deposition (PVD or CVD), electrophoretic deposition (EPD), *etc.*[7-9] Among them, atmospheric plasma spraying (APS) technology has unique advantages due to its high deposition rate and low cost. However, the YSZ coating prepared by the APS process is stacked by the layered splats from melted or semi-melted particles, which makes it challenging to obtain a uniform and defect-free microstructure [10-14]. Other vacuum filming technology like PVD or CVD can manufacture high-quality YSZ coating but needs long preparation time and high investment cost [15-19].

To solve the problem, the very low-pressure plasma spray (VLPPS) process has emerged by adding a vacuum tank based on the APS, which can significantly expand the plasma jet and increase the jet velocity under very low pressure (<800 Pa). As a result, the injected YSZ powder can be heated more uniformly, and its impact velocity can be increased to 2-3 times the speed of sound. This improvement will significantly enhance the densification of the YSZ coating and maintain a relatively high deposition efficiency [20-25]. However, since the low-pressure environment weakens the heating capacity of the plasma jet, using a conventional low-power plasma torch (F4-VB, Oerlikon metco) is difficult to obtain a YSZ coating with a density higher than 95% [26]. Hence, previous studies chose a higher energy plasma torch with a maximum power of 180 kW (O3CP, Oerlikon metco) to manufacture the YSZ coating, which achieved the density of coating to 99.56% [27-29].

Since the preparation cost of using the O3CP torch is still too high, this study designed a new axial bi-cathode plasma torch for VLPPS process based on two F4-VB plasma guns, which realized a maximum spraying power of 81 kW and an axial feeding of YSZ powder. By using this torch, a dense and uniform YSZ coating with a mixed microstructure was fabricated under 6 mbar. The melting state, microstructure, phase composition, and mechanical property of the YSZ coating were carefully investigated.

2. Experimental process

Fig.1 displays the sketch map of the bi-cathode plasma torch and the VLPPS system. To achieve higher plasma power and axial powder feeding, the designed torch combined two conventional DC plasma guns (F4-type, Oerlikon metco) by sharing one anode. Specifically, the bi-cathode plasma torch retained the rear body (1), the cathode (2), and the insulator (3) of the F4-type gun, but reformed its middle body (4), anode (5), and anode holder (6,7). Two separated F4-type cathodes (2) were placed on both sides of the powder injector (8) with an angle of 17°. The anode (5) was installed

above the middle body (4) as a common nozzle. Since the relative location between the cathodes (2) and anode (5) is critical to generate plasma arc and maintain plasma symmetry, the machining size of the middle body (4) was precisely controlled. With the plasma gas injected from the bottom of cathodes (2), two individual plasma jets would be formed, and then merged into one stream through the internal passage of the anode (5). Afterward, the YSZ particles could be axially delivered into the converging jet center via the powder injector (8), even at very low pressure. Since the jet center had the highest plasma temperature and velocity, the sprayed YSZ particles could be sufficiently heated and accelerated. For operating this torch, two power supplies and control cabinets were adopted, which allowed the spraying power to reach 81kW. For spraying experiment, 7~8 wt% YSZ agglomerated powder (Metco 6700, Oerlikon metco) with an average particle size of 10 μm was used as the spraying material. The carbon steel plates were chosen as the substrate, with a 150 μm CoNiCrAlY bond coating (Amdry 9951, Oerlikon metco) deposited by an APS process. The detailed spraying parameters are shown in Table 1. In order to maximize heating and acceleration of YSZ powder to improve coating density, a minimum pressure of 6mbar and a maximum power of 81kW had been adopted. Besides, a large amount of He gas was used for spraying because it has higher thermal conductivity and can form a narrower plasma jet than Ar gas. As a result, the YSZ powder with a high flying speed was easier to be melted/vaporized, which was conducive to coating densification.

For characterization, the optical emission spectrometer (OES, Jobin-Yvon) equipped with a CCD detector was adopted to analyze the species in the plasma jet during the spraying process. The surface and cross-section morphology of the YSZ coating was detected by the scanning electron microscope (SEM, JSM-7800F). Based on five different SEM images, the average coating porosity was estimated by using the ImageJ software [30, 31]. The phase composition of the powder and as-sprayed coating was characterized by the X-ray diffraction (XRD, Rigaku) with a Cu $K\alpha$ radiation. The mechanical property of the coating was identified by the nanoindentation (Hysitron) with a maximum load of 10 mN. The loading/unloading rate, holding time, and temperature was 20 mN/min, 10 s, and 25 $^{\circ}\text{C}$, respectively [32].

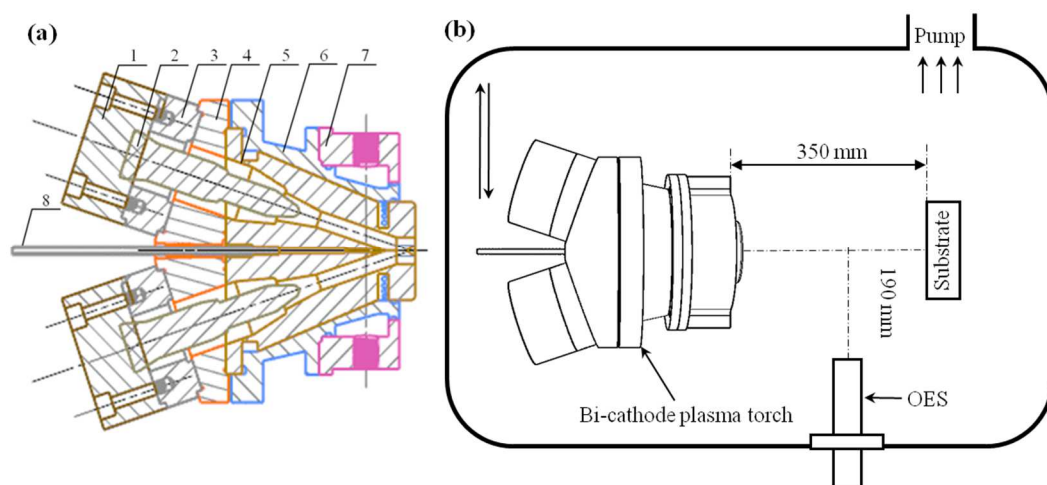


Fig.1 The sketch map of (a) axial plasma torch: 1-rear body, 2-cathode, 3-insulator,

4-middle body, 5-anode, 6-anode holder bottom, 7-anode holder top, 8-powder injector;
(b) VLPPS system.

Table 1 Parameters of the VLPPS.

Parameters	Value
Pressure (mbar)	6
Current (A)	1400
Power (kW)	81
Plasma gas Ar/He/H ₂	6:10:1
Total gas flow rate (slpm)	170
Powder feeding rate (g/min)	2
Spraying distance (mm)	350
Torch speed (mm/s)	200

3. Results and discussions

Fig.2 shows the OES patterns of the plasma jet without and with YSZ powder. It can be found that the spectral line of the plasma gas was mainly located at 600-900 nm, while that of the YSZ powder was between 500-600 nm. The appearance of the YSZ line demonstrated the axial bi-cathode plasma torch had evaporated the YSZ powder. However, the intensity of the YSZ line was much lower than that of the plasma gas, indicating that only part of the YSZ powder was vaporized. The result was logical since the detected position is at 100 mm, so the heating time for the YSZ powder was extremely short. Von Niessen et al. [33] reported a similar result that low intensity of the YSZ spectral line vaporized a minor amount of YSZ powder and produced a splat-like coating.

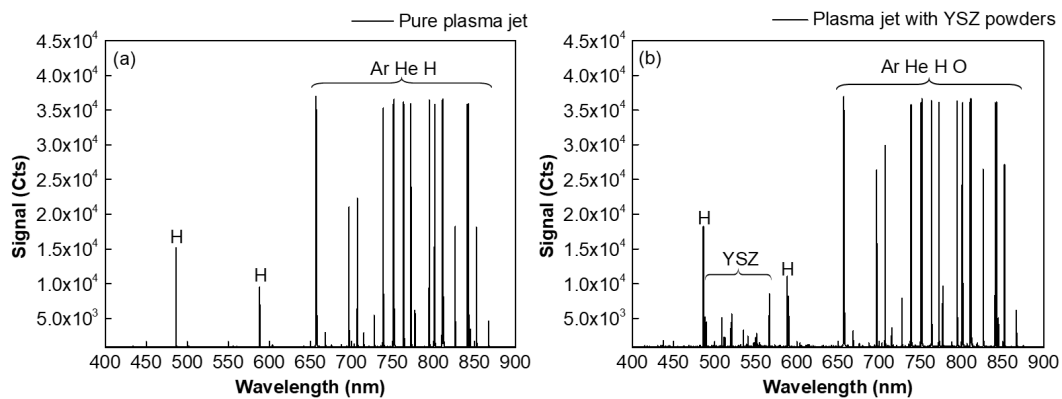


Fig. 2 The OES patterns of the plasma jet (a) without and (b) with YSZ powder.

Fig.3 (a-a') presents the SEM image of the YSZ coating on the substrate front. It reveals that the YSZ coating possessed a mixed microstructure, which was composed of the unmelted powder, melted splats, and fine clusters. The hybrid microstructure was primarily due to the diverse powder sizes, and deficient heating caused by the large

gas flow. As the agglomerated YSZ powder was delivered into the plasma center, the high-temperature plasma jet with high velocity would break it up and reduce the size to the sub-micrometer level. However, the broken time for different-sized powder was not the same. The small agglomerated YSZ powder could be rapidly fragmented and melted to form the splat, or even be vaporized to the cluster. But for the big agglomerated YSZ powder, more time was needed for the fragmentation. When the fragmentation finished, the YSZ powder was no longer in the hot region of the plasma jet due to the large gas flow. Therefore, the unmelted powder was formed and eventually remained in the coating. Fortunately, the image analysis showed that the hybrid microstructure gave the YSZ coating a low porosity of 2.7%, which was superior to that of 5.8% prepared by APS [34]. Moreover, it is worth noting that the YSZ coating with a columnar-like microstructure can also be observed on the substrate side, as shown in Fig. 3 (b-b'). Since the liquid splats and big unmelted powder were difficult to adhere to the substrate side, the YSZ coating consisted almost entirely of the clusters, which derived from the fine unmelted powder and the vaporized phase. It proved that some YSZ powder was indeed vaporized by the plasma jet.

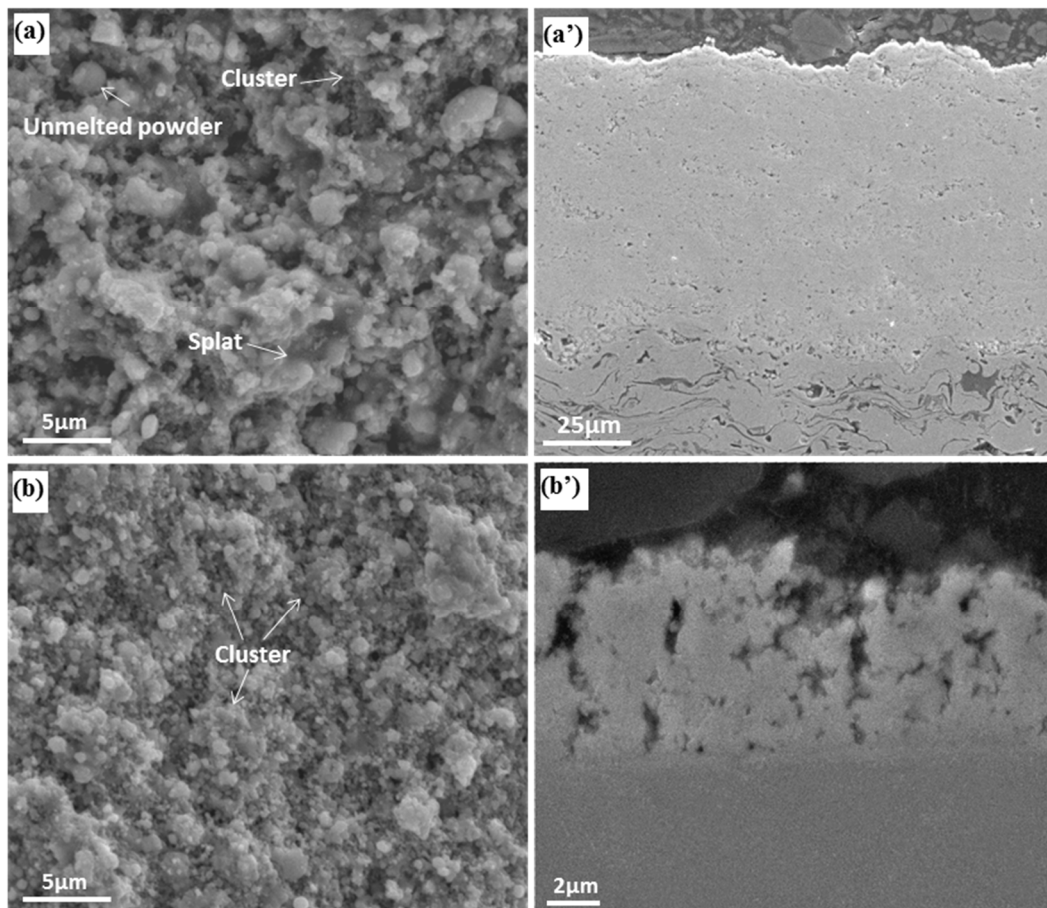


Fig.3 The surface and section morphologies of the YSZ coatings obtained on the substrate front (a-a') and substrate side (b-b').

Fig.4 displays the phase composition of the YSZ powder and the resulting coating. The YSZ powder consisted of a monoclinic zirconia phase (M-ZrO₂) and a cubic yttria

phase (C-Y₂O₃), while the YSZ coating produced a new tetragonal-prime zirconia phase (T'-ZrO₂) beside them. According to the phase diagram of YSZ, when the YSZ powder was fully melted by the plasma jet, the M-ZrO₂ would first be converted to the cubic zirconia phase (C-ZrO₂). Then the C-ZrO₂ impinged on a colder substrate or previously stacked splats, and a high quenching rate would result in rapid solidification of C-ZrO₂ in a non-equilibrium state and produce a medium-temperature phase of T'-ZrO₂ with high Y₂O₃ content. Therefore, the content of T'-ZrO₂ in the YSZ coating was a sign of the melting degree of YSZ powder. To further study this relationship, the content of T'-ZrO₂ was estimated based on the RIR method [35, 36]. The calculation result showed that the YSZ coating contained 40% T'-ZrO₂, indicating the YSZ powder was partially melted by using the designed torch. Compared with the full T' phase YSZ coating by using other higher power O3CP torch (≥ 120 kW) [27, 37], the T'-ZrO₂ content acquired in this study was not high enough. The reason is that the bi-cathode plasma torch adopted a limited plasma power (81kW) and a much larger gas flow (170 l/min), leading to the refractory YSZ powder did not have sufficient energy and time to be fully melted. Therefore, in order to better melt or evaporate the YSZ powder, the power and structure of the bi-cathode plasma torch need to be further improved.

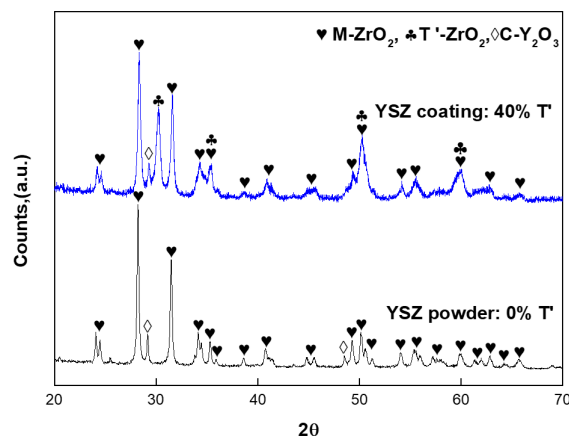


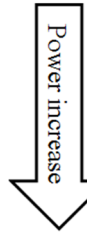
Fig.4 The XRD pattern of the YSZ powder and as-sprayed coating.

Table 2 compares the mechanical properties of YSZ coatings in this study and other literature. For the plasma spray process, the microhardness (H) and elastic modulus (E) of YSZ coating in this work are 11.7 ± 0.6 GPa and 167.8 ± 4 GPa, respectively. These values are better than the results of Ghasemi et al.[38] and He et al.[20], who separately adopted the F4-type plasma torch for APS and VLPPS. It can be attributed to the fact that the bi-cathode plasma torch used in this study has a higher plasma power and faster plasma speed, making the injected YSZ powder melt more fully and combine more tightly. Gao et al. [27] used a more powerful MC-100 torch to deposit the YSZ coating, promoting the heating of YSZ powder to be vaporized, but the mechanical property obtained is still lower than that in current work. This is because the YSZ coating deposited by vapor phase had a loose columnar microstructure rather than a dense one. It can be also used to explain that when Shao et al. [28] adopted the same high-power O3CP torch to fabricate compact YSZ coating,

the coating with densest structure had the best mechanical property. In other words, to enhance the mechanical property of YSZ coating, increasing the density of the coating structure may be more effective than simply improving the heating of the powder. For other preparation technologies, such as EB-PVD [39, 40], the mechanical property of YSZ coating in this paper is obviously better because the coating structure is denser. To achieve this level, the EB-PVD YSZ coatings with columnar microstructures require high-temperature heat treatment. It will further increase the complexity of EB-PVD, since its deposition efficiency is originally much lower than that of plasma spraying.

Table 2 Mechanical properties of the YSZ coatings obtained in this work compared with literature.

Author	Process	Microhardness	Elastic modulus
Ghasemi et al. [38]	APS, F4-MB torch	5.5~16.0 GPa	85.2~202.9 GPa
He et al. [20]	VLPPS, F4-VB torch	10.4±0.9 GPa	-
Present work	VLPPS, Bi-cathode torch	11.7±0.6 GPa	167.8±4 GPa
Gao et al. [27]	PS-PVD, MC-100 torch	9.0 GPa	110.9 GPa
Shao et al. [28]	PS-PVD, O3CP torch	16.6±0.6 GPa	234.3±10.1 GPa
Guo et al. [39]	EB-PVD	3.8 GPa	53.3 GPa
Chen et al. [40]	EB-PVD	8.45 GPa	153.15 GPa



4. Conclusions

In this study, the axial bi-cathode plasma torch with a maximum power of 81 kW was designed to prepare the YSZ coating under 6 mbar. By characterizing the vaporization state, microstructure, phase composition and mechanical property of the coating, the following conclusions can be drawn:

(1) Most of the YSZ powder was melted by the bi-cathode plasma torch, and part of it was even vaporized. Meanwhile, 40% of the M-ZrO₂ phase in the YSZ powder was converted to the T'-ZrO₂ phase, indicating the powder was melted well.

(2) The dense and uniform YSZ coating with a porosity of 2.7% was obtained, which was deposited with the form of unmelted particles, melted droplets, and vapor clusters.

(3) The H and E of the YSZ coating were 11.7±0.6GPa and 167.8±4GPa, respectively. To enhance the mechanical property of YSZ coating, increasing the density of the coating structure may be more effective than simply improving the heating of the powder.

(4) Using the bi-cathode plasma torch at low pressure produced the YSZ coating with high density and mechanical property, which demonstrated that it has excellent potential for the preparation of SOFC electrolyte.

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