Plasma-Sprayed $(\text{Bi}_2\text{O}_3)_{0.705}(\text{Er}_2\text{O}_3)_{0.245}(\text{WO}_3)_{0.05}$ Electrolyte for Intermediate-Temperature Solid Oxide Fuel Cells (IT-SOFCs)

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Abstract Stabilized bismuth oxide material with fluorite structure ($\delta\text{-Bi}_2\text{O}_3$) has been studied as a promising electrolyte material for intermediate temperature solid oxide fuel cells (IT-SOFCs) due to its high oxygen ion conductivity in mediate temperature. Especially, the ternary system $\text{Bi}_2\text{O}_3\text{-Er}_2\text{O}_3\text{-WO}_3$ is widely concerned for its high ionic conductivity and thermal stability. In this study, regarding its low melting point, the possibility to deposit dense $\text{Bi}_2\text{O}_3\text{-Er}_2\text{O}_3\text{-WO}_3$ ($(\text{Bi}_2\text{O}_3)_{0.705}(\text{Er}_2\text{O}_3)_{0.245}(\text{WO}_3)_{0.05}$, EWSB) electrolyte by plasma spraying was examined. It was confirmed that the sintered EWSB bulk presents a high ion conductivity of 0.34 S cm^{-1} at 750°C and excellent stability that indicates no structure transformation and conductivity degradation after annealing at 600°C for 1000 h. The phase structure and cross-sectional microstructure of plasma-sprayed EWSB were characterized by XRD and SEM. Results showed that the as-plasma-sprayed EWSB presents a dense microstructure with well bonded lamellae. The XRD showed the formation of EWSB with δ -phase and a trace of β -phase, while the β -phase disappeared after annealing at 750°C for 10 h. The deposited EWSB electrolyte presented the excellent ionic

conductivity of 0.26 S cm^{-1} at 750°C which can be directly applied to SOFC at intermediate temperature.

Keywords Bismuth oxide · EWSB electrolyte · ionic conductivity · IT-SOFC · plasma spraying

Introduction

Solid oxide fuel cells (SOFCs) are the promising energy conversion devices that directly convert chemical energy to electrical energy via an electrochemical reaction for their high efficient energy conversion up to 65% (Ref 1). As the solid-state cells, SOFCs consist of the cathode (reduce oxygen gas from air to oxygen ions), electrolyte (transport oxygen ions), and anode (oxygen ions react with fuel and release electrons to the external circuit.). The major technical issue that limits the commercialization of SOFC is the high operating temperature resulting in the high system cost. The lower operating temperature of SOFC reduces thermal and chemical stresses, which means enhanced system stability and a wider choice range of inexpensive materials (Ref 2). Over the past decades, many researchers were concerned with the development of high conductive electrolyte materials at low temperatures for lowering the operating temperature (Ref 3). The most widely used electrolyte material for SOFCs is zirconia-based materials, typically yttria-stabilized zirconia (YSZ) which exhibits excellent stability. However, YSZ is not suitable to be used at low operating temperatures as the electrolyte of SOFCs for its low ionic conductivity at temperatures lower than 800°C (Ref 4). As reported, bismuth oxide systems ($\delta\text{-Bi}_2\text{O}_3$) have been demonstrated to exhibit superior oxide ion conductivity (Ref 5–8). The conductivity of isovalent rare earth oxide stabilized Bi_2O_3 (SBO) is 1–2 orders of

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magnitude higher than that of YSZ (Ref 9). Among the SBO materials, the 20% Er_2O_3 stabilized Bi_2O_3 (ESB) exhibits the highest ionic conductivity (Ref 1). Nevertheless, it was proved that “the stabilized δ -phase of ESB material” is a metastable quenched phase, and the transformation of the fluorite δ -phase occurs at about 600 °C accompanying the degradation of conductivity and change of volume (Ref 8). System Bi_2O_3 - Er_2O_3 - WO_3 in a narrow limited compositional range has been investigated to be a truly stabilized δ -phase without any degradation of ionic conductivity. As reported, in contrast to remarkable aging degradation of SBO materials, $(\text{Bi}_2\text{O}_3)_{0.72}(\text{Er}_2\text{O}_3)_{0.22}(\text{WO}_3)_{0.06}$ exhibits only a degradation less than 1.3% at 600 °C after 830 h, while $(\text{Bi}_2\text{O}_3)_{0.705}(\text{Er}_2\text{O}_3)_{0.245}(\text{WO}_3)_{0.05}$ shows no time-dependent degradation at 600 °C during 1100 h (Ref 10).

Up to now, various methods have been developed to prepare bismuth-based electrolytes. The conventional methods mainly based on ceramic powder sintering such as sol-gel (Ref 11), screen printing (Ref 12), dip coating (Ref 13) have been widely used. The electrolyte particles attached to the electrode required subsequent high-temperature sintering to obtain a dense structure (Ref 14, 15). Subsequently, the interface reaction between the electrolyte and the electrode under high temperatures occurs which affects the cell performance (Ref 16). Especially, the high sintering temperature is not suitable for bismuth-based materials with low melting points (lower than 1000 °C). Thus, the low temperature processing methods, such as direct current (DC) magnetron sputtering (Ref 17), vapor deposition (Ref 18), and pulsed laser deposition (PLD) (Ref 19) were investigated for the deposition of electrolyte. Although a thin and dense electrolyte can be prepared by the above-mentioned methods, the process is complicated and expensive. Plasma spraying technology has significant advantages, such as low cost and high film-forming efficiency, one-step forming, which can be used for the preparation of large-size and complex-shaped SOFC. As a cost-effective electrolyte preparation process, it has promising potential in the manufacturing of efficient high-performance SOFC. Based on the previous investigations, high-performance YSZ (Ref 20) and $\text{La}_{0.8}\text{Sr}_{0.2}\text{Ga}_{0.8}\text{Mg}_{0.2}\text{O}_{3-\delta}$ (LSGM) (Ref 21) electrolytes can be fabricated by atmospheric plasma spraying. The major challenge is that the limited interlayer bonding ratio and high porosity in the coating make it difficult for plasma sprayed ceramic coatings to be directly applied to the SOFCs. The low melting point of bismuth materials makes it possible to fabricate dense electrolytes via the plasma spraying method. Based on the critical bonding temperature theory reported recently (Ref 22), for the ceramic material with a melting point lower than 1500 °C, the dense coating with sufficiently bonded lamellae can be directly fabricated by

the atmospheric plasma spraying method without any preheating of the substrate (Ref 23). As in our previous work, the dense Y_2O_3 stabilized Bi_2O_3 electrolyte has been prepared by plasma spraying (Ref 24). However, the Bi element evaporates preferentially due to the higher vapor pressure of Bi_2O_3 during plasma spray process. The higher doped concentration of rare earth oxide from the original powders affects the ionic conductivity of bismuth-based deposits. There was no investigation concerning the influence of element evaporation and rapid heating quenching processes during spraying on the performance of ternary bismuth-based electrolyte by plasma spraying yet.

During plasma spraying of multiple components of ceramics, the preferential vaporization of the component with high vapor pressure occurs which results in a deviation of deposit composition from its starting powders (Ref 25). Moreover, a strong size effect on the preferential vaporization was observed to show that smaller powder particles present a higher loss of the component with a high vapor pressure (Ref 21, 26). As a result, the deposit plasma-sprayed with a wide particle size distribution may present a nonuniform distribution of different components. However, such inhomogeneity of compositions may be recovered by post-deposition annealing (Ref 27).

In this work, the $(\text{Bi}_2\text{O}_3)_{0.705}(\text{Er}_2\text{O}_3)_{0.245}(\text{WO}_3)_{0.05}$ (EWSB) electrolyte was fabricated by plasma spraying to investigate the crystal structure, microstructural features, and ionic conductivity of deposited EWSB materials. The effect of the annealing treatment on the structure, element distribution, and ionic conductivity of the EWSB deposit was also examined.

Experimental Procedure

Materials and Coating Deposition Process

The high purity nano (<200 nm) oxide powders of Bi_2O_3 (99.99% purity, Ningbo Begaer), Er_2O_3 (99.99% purity, Zhengde Rare Earth), and WO_3 (99.99% purity, Ningbo Begaer) were used to prepare the EWSB powders with the stoichiometric ratios of $(\text{Bi}_2\text{O}_3)_{0.705}(\text{Er}_2\text{O}_3)_{0.245}(\text{WO}_3)_{0.05}$ through solid state reaction. The starting mixtures of the materials were milled in ethanol for 20 h. Then 5 wt.% polyvinyl alcohol solution was added into the dried powder mixtures to enhance the bonding between the powder particles. The mixtures were further reground in an agate mortar for 10 min. The powders were sintered at a temperature of 800 °C under ambient atmospheric conditions for 20 h and were slowly cooled down in the furnace. The as-prepared powders were crushed and sieved through 200 and 400 meshes to obtain the powders in a size range of 37.5 to 75 μm .

Figure 1 shows the morphology, cross-sectional microstructure, and XRD pattern of synthesized EWSB powder. As shown in Fig. 1(a), the powder presents an irregular shape with small size particles attached to the surface of powders. As shown in Fig. 1(b), the powder shows a dense structure although some small pores are present in the powder. The XRD pattern of the EWSB powders is shown in Fig. 1(c), and it can be clearly seen that the EWSB powders have a single δ -phase (PDF #40-0320) and no impurity phase was observed.

Fig. 1 (a) Morphology of EWSB powders; (b) Cross-sectional morphology of EWSB powder; (c) XRD pattern of EWSB powder

Figure 2 illustrates the thermal stability of sintered EWSB and ESB prepared by powders synthesized through solid state reaction in terms of ionic conductivity against annealing time at a constant temperature of 600 °C. Although the ESB material shows the decay of conductivity at 600 °C after about 300h resulting from the transformation of δ -phase, the conductivity test of EWSB shows the long-term stability of δ -phase, since no degradation was observed during 1000 h at 600 °C in the conductivity. The synthesized EWSB material has been proved to be stable for a long time duration.

EWSB deposit was plasma-sprayed using a commercial plasma spray system (80 kW, Jiujiang, China). Typical plasma spray parameters are listed in Table 1. The plasma torch was operated using Ar/H₂ gases at a plasma arc power of 25 kW. The plasma torch traverse speed over the substrate was 400 mm/s. NaCl pellets with a diameter of 20 mm and a thickness of 2 mm were used as the substrate to obtain free-standing EWSB samples for the measurement of the ionic conductivity.

In order to make a comparison with the deposit, 2.0 g EWSB powders were pressed into pellets with 13 mm in diameter and sintered at a temperature of 800 °C for 10 h. The as-sprayed EWSB deposits are also annealed in air at 750 °C for 10 h to investigate the influence of the subsequent annealing.

Characterization Methods

The x-ray diffraction analysis (XRD, D8 ADVANCE) was performed to characterize the crystal structure of EWSB samples. The microstructure of EWSB samples was characterized by scanning electron microscopy (SEM, Hitachi, S-4800). The porosity of EWSB samples was estimated by image analyzing method using SEM images taken in the BEI mode from the polished cross sections. The energy-dispersive spectroscopy (EDS) was used for element

Fig. 2 Ionic conductivity as a function of time at 600 °C for EWSB and ESB

Table 1 Plasma spray parameters

Spray parameter	Unit	Value
Plasma arc power	kW	25
Spray distance	mm	80
Primary plasma gas (Ar)	L·min ⁻¹	45
Auxiliary plasma gas (H ₂)	L·min ⁻¹	2

quantitative analysis of the samples. The conductivities of electrolytes were obtained by electrochemical impedance spectroscopy analysis. The silver paste was adopted as the electrode and coated on both sides of the sample symmetrically. The impedance measurements were performed in a temperature range from 350 to 750 °C on the Solatron Electrochemical Analysis Station (SI 1260/1287) in a frequency range from 0.1 to 100 kHz and 25 mV AC bias.

Results and Discussion

Microstructure of EWSB Samples

To examine the interlamellar bonding state of the EWSB deposit, the sprayed deposit was fractured from cross section and its morphology was compared with those of both the annealed EWSB deposit and sintered EWSB sample. Figure 3(a) and (b) shows the cross-sectional morphology of the fractured EWSB deposit. The fractured surface of the as-sprayed EWSB deposit in Fig. 3(a), (b) shows that the interlamellar bonding feature of the EWSB deposit is obviously different from the conventional ceramic spray coatings. The fractured EWSB deposit shows a morphology analogous to sintered bulk (Fig. 5a, b), indicating the formation of dense deposit with well-bonded lamellae. Several pores and voids were observed as shown in Fig. 3(a), (b). The global pores in the size of several micrometers are possibly retained from the gas phase enclosed in powder during the solidification process (Ref 28). The voids with irregular shapes are resulting from incomplete filling and infiltration during the spreading of molten droplets. Figure 4(a), (b) shows the morphology of the fracture surface of the annealed deposit. It can be recognized that the annealed EWSB also presents a similar morphology with the press-sintered EWSB bulk (Fig. 5(a), (b)) although a few larger pores were present on the annealed deposit than the sintered bulk. Such a feature can be attributed to the characteristic deposition feature of ceramic of a low melting point during plasma spraying (Ref 23).

Figure 6, 7 and 8 show the cross-sectional microstructures of the as-plasma-sprayed EWSB deposit, annealed

Fig. 3 Morphology of the fractured plasma-sprayed EWSB deposit: (a), and (b)

EWSB deposit and sintered EWSB sample, respectively. As shown in Fig. 6(a), (b), the as-sprayed deposit shows a dense microstructure with a well-bonded lamellar interfaces. The estimation yielded a porosity of $\sim 2.4 \pm 0.6\%$. The pores and cracks are observed on the cross section of polished EWSB deposit in Fig. 6(b). Some pores present a nearly spherical shape which is consistent with that observed from the fractured coating surface (Fig. 3(a), (b)). The cracks with several micrometers long possibly resulted from the accumulated quenching stress during rapid cooling of multiple splats after solidification. Localized spalling due to inter-section of cracks during the sample preparation process may appear as pores. The annealed deposit in Fig. 7(a), (b) shows a microstructure without the cracks, although larger pores even up to $\sim$ ten micrometers were present possibly due to coalescence of pores and voids. The press-sintered EWSB bulk as shown in Fig. 8(a), (b) shows the dense microstructure with pores of several micrometers.

As in previous work, the bismuth evaporates faster from the surface of the molten splat than rare earth elements during spraying, resulting in less bismuth which is consistent with the different concentrations of cation ions. To

Fig. 4 Morphology of the fractured annealed plasma-sprayed EWSB deposit: (a), and (b)

investigate the influence of bismuth evaporation on the distribution of elements, as shown in Fig. 9(a), a close examination into the polished cross-section of the deposit by backscattered electron imaging with SEM reveals the existence of the splat in a relatively darker gray contrast with a thickness of $\sim 0.3 \mu\text{m}$. The distributions of Bi, Er, W elements along the line across the above mentioned splat by EDS are shown in Fig. 9(b). The results indicate that this splat has relatively low Bi content and high Er content. The inhomogeneous evaporation of Bi is attributed to the different sizes of original powders, especially small particles adhered to the surface of powders. Moreover, no splat with a gray contrast was observed in the annealed EWSB deposit as shown in Fig. 10. This fact indicates that the diffusion could take place during annealing to homogenize the microstructure of the EWSB deposit. As a result, although the EWSB dense electrolyte with inhomogeneous distribution of elements was fabricated by atmospheric plasma spraying, post-spray annealing results in the EWSB deposit with uniform distribution of elements.

Fig. 5 Morphology of the fractured sintered EWSB sample: (a), and (b)

Phase Structures of EWSB Samples

The XRD pattern of the EWSB deposit shown in Fig. 11 reveals the additional small peaks of tetragonal β -phase in addition to predominantly the peaks of fluorite structure δ -phase. As can be seen in Fig. 6, after the annealed at 750°C , the small peaks of the tetragonal β -phase disappeared and only peaks of the high degree of crystallization δ -phase were observed. In addition, the peaks of δ -phase were also observed in the EWSB deposit when the deposition temperature (the substrate temperature during the spraying process) increased to 350°C . Thus, the metastable β -phase disappeared after annealing at 750°C due to the redistribution of Bi through Bi diffusion during annealing. Therefore, it can be considered that the formation of β -phase is attributed to the evaporation of Bi resulting in low Bi content metastable phase. In addition, compared with the sintered EWSB bulk, the peaks of fluorite structure δ -phase in the deposit shift to the right slightly as the lattice constants are reduced due to loss of Bi.

Fig. 6 Cross-sectional microstructures of free-standing plasma-sprayed EWSB deposit: (a), and (b)

Ionic Conductivity of EWSB Deposit

The conductivity of the as-sprayed EWSB electrolyte versus $1000/T$ (K) measured under ambient atmosphere is shown in Fig. 12, in comparison with both the annealed and sintered samples. It can be seen that the conductivity increases with increasing temperature due to the increased ion vacancies and the thermal vibrations of the ceramic lattice. The ionic conductivity data of the three samples are shown in Table 2 for typical temperatures. The ionic conductivity values of the as-sprayed EWSB deposit at the temperatures of 750 °C and 550 °C are $2.6 \times 10^{-1} \text{ S cm}^{-1}$ and $1.7 \times 10^{-2} \text{ S cm}^{-1}$, respectively. Although the deposit presents good ionic conductivity, compared to sintered bulk with the ionic conductivity of $3.4 \times 10^{-1} \text{ S cm}^{-1}$ at 750 °C and $3.0 \times 10^{-2} \text{ S cm}^{-1}$ at 550 °C, the ionic conductivity of the as-sprayed EWSB deposit only accounts for 73% at 750 °C and 43% at 550 °C of that of the sintered EWSB bulk. These results can be attributed to the low density and high concentration of Er and W elements of EWSB deposit. Although the as-sprayed EWSB deposit shows a dense structure, the density of the deposit is still lower than sintered bulk. On the other hand, a higher

Fig. 7 Cross-sectional microstructures of annealed EWSB deposit: (a), and (b)

content doping leads to a lower conductivity according to the previous results (Ref 24). According to thermogravimetric analysis results on the green powders, the mass loss due to the volatilization of bismuth oxide during the synthesis process was confirmed to be negligible (Ref 29). Nevertheless, for the EWSB deposit, as shown by EDS results in Table 3, the concentration of Bi decreased from 72.4% in the powder to 69.8% in the deposit accompanying the increase of Er and W contents from 24.8 to 26.5 and 2.8 to 3.7%, respectively. The conductivity of the EWSB deposit can be improved by adjusting the doped ratio of Er_2O_3 and WO_3 .

As shown in Fig. 12, the ionic conductivity curves of three EWSB electrolytes all show the knee at about 600 °C which indicates the change of the activation energies of the EWSB electrolyte. The change of activation energies at about 600 °C suggests that the electrolyte sample undergoes an order-disorder transition of the oxygen sublattice. In the low temperature region under 600 °C, the oxygen sublattice of EWSB is ordered, the composition has no significant influence on the thermally activated passage of oxygen ions through the lattice (Ref 5). In the high temperature region above 600 °C, the oxygen lattice of EWSB

(a)

(b)

Fig. 8 Cross-sectional microstructures of sintered EWSB bulk : (a), and (b)

is disordered analog to pure δ - Bi_2O_3 . Table 4 shows the activation energies E_a calculated from the data in Fig. 12. As shown in Table 4, in the high temperature range (over 600 °C), the activation energy of deposit (0.81 eV) is higher than that of sintered bulk (0.74 eV), whereas those in the low temperature range were almost constant. The higher activation energies of deposit at high temperatures indicate that the higher content of doped Er, W content in EWSB electrolyte enhance the stability of order oxygen sublattice. With increased doped element content of EWSB electrolyte, the concentration of the mobile oxygen ions strongly decreases, part of order oxygen vacancies in the sublattice is maintained to a higher temperature.

As shown in Fig. 12 the EWSB deposit after annealing at 750 °C for 10 h showed a conductivity similar to the as-sprayed EWSB deposit at the corresponding temperature, although the microstructure and phase structure between the as-sprayed deposit and the annealed deposit is different. The reason is that during the measurement process of conductivity, the microstructure and phase structure changed during the heating process of the measurement. As can be seen in Fig. 13, the microstructure of the polished cross-section of the EWSB deposit after the measurement of the

(a)

(b)

Fig. 9 (a) Close examination of cross-sectional polished plasma-sprayed EWSB deposit; (b) EDS line scans Bi, Er, W across the region as marked in (a)

Fig. 10 Close examination of cross-sectional polished annealed EWSB deposit

ionic conductivity shown in Fig. 13(a)–(c) and XRD pattern shown in Fig. 13(d) are similar to those of the annealed deposit. The distribution of elements becomes uniform and no striped splat region with a gray contrast was observed as shown in Fig. 13(a)–(c).

Fig. 11 The XRD patterns of EWSB deposit, annealed EWSB deposit, and sintered EWSB bulk

Fig. 12 The temperature dependence of the ion conductivity of sintered EWSB deposit, annealed EWSB deposit, and sintered EWSB bulk

Table 2 The ionic conductivity of the EWSB samples at 750 and 550 °C

Sample	Ionic conductivity σ , S cm ⁻¹	
	750 °C	550 °C
Deposit	2.6×10^{-1}	1.7×10^{-2}
Annealed deposit	2.7×10^{-1}	2.1×10^{-2}
Sintered bulk	3.4×10^{-1}	3.0×10^{-2}

Table 3 Bi, Er, W percentage of EWSB powder and deposit

Element/%	Powder	Deposit
Bi	72.4	69.8
Er	24.8	26.5
W	2.8	3.7
Total	100	100

Table 4 Activation energy of EWSB samples

EWSB	Activation energy, eV	
	>600 °C	<600 °C
Deposit	0.81	1.24
Annealed deposit	0.81	1.22
Sintered bulk	0.74	1.23

The results proved that the sprayed EWSB deposit can be directly applied to the SOFC at the intermediate operating temperature without any necessity of subsequent annealing.

Conclusions

The fracture surface of the as-sprayed EWSB revealed a structure similar to that of sintered EWSB. A dense EWSB deposit with sufficiently bonded lamellae was plasma-sprayed using dense powder particles. The results showed the preferable Bi evaporation loss of small powder particles led to the formation of thin EWSB splats with low Bi content. A trace of tetragonal β -phase was observed in the as-sprayed deposit besides fluorite structure δ -phase. The post-spray annealing and in-situ annealing during the heating process for ionic conductivity measurement can eliminate the inhomogeneity of microstructure and tetragonal β -phase in the as-sprayed EWSB deposit. Although the conductivity of the as-sprayed EWSB was lower than the ion conductivity of the sintered bulk at the corresponding temperature, the as-sprayed EWSB deposit presented a high ionic conductivity of 2.6×10^{-1} S cm⁻¹ and 1.7×10^{-2} S cm⁻¹ at temperatures of 750 °C and 550 °C, respectively. The EWSB deposit also showed higher activation energies than sintered EWSB at high temperature which is related to the high doped element content.

Fig. 13 Cross-sectional microstructures of EWSB deposit after ionic measurement (a), (b), and (c); the XRD patterns of EWSB deposit after ionic measurement (d)

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