High ionic conductivity of Lu$_2$O$_3$–TiO$_2$ co-doped Bi$_2$O$_3$ ceramics

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Abstract
In this study, a promising electrolyte material Bi$_{0.76}$Lu$_{0.16}$Ti$_{0.08}$O$_{1.5}$ (abbreviated as ‘8T16LSB’) with high oxygen ion conductivity was obtained by co-doping with Lu$_2$O$_3$ and TiO$_2$ into Bi$_2$O$_3$. The phase composition, microstructure, and conductivity of TLSB ceramics were respectively investigated by x-ray diffraction, scanning electron microscopy (SEM), and electrochemical impedance spectroscopy (EIS). XRD results exhibited that the sintered TLSB ceramics with an unchanged total doping concentration of 24 mol.% existed as a cubic fluorite structure. SEM results showed that the TLSB pellets were relatively dense. The relationship between temperature and conductivity showed that the 8T16LSB ceramic exhibited the highest conductivity of 0.56 S cm$^{-1}$ at 700 °C, which was higher than the value of 0.37 S cm$^{-1}$ for the representative single-doping Er$_{0.4}$Bi$_{1.6}$O$_3$ (abbreviated as ‘20ESB’). The results certified that the electrochemical performance of Bi$_2$O$_3$ was promoted by the co-dopant of Lu$^{3+}$ and Ti$^{4+}$.

1. Introduction

The development of intermediate temperature solid oxide fuel cells (IT-SOFCs) has attracted increasing attention for the direct conversion of chemical energy to electrical energy [1]. The electrolyte material has a significant impact on the electrical performance of the single cell [2]. According to previous investigations, fluorite and perovskite-type structural materials are the most frequently-used electrolytes for IT-SOFCs [1, 3–6]. While the traditional electrolyte YSZ usually works well at higher temperatures > 700 °C; this results in high-cost cells and also hinders the practical application of SOFCs [7, 8]. Therefore, it is necessary to develop electrolyte materials with a high ionic conductivity working that function at low and intermediate temperatures [9, 10].

Among the several possible electrolytes, δ-Bi$_2$O$_3$ with the fcc structure is considered as one of the most promising oxide ion conductors [11, 12]. The ionic conductivity of δ-Bi$_2$O$_3$ is about two orders of magnitude higher than that of YSZ, which is currently served as the SOFC electrolyte. Furthermore, δ-Bi$_2$O$_3$ exhibited high oxygen ion conductivity because of its high randomly distributed oxygen vacancy concentration and the weak Bi-O bond which resulted in high oxygen mobility [13]. Therefore, it is feasible that δ-Bi$_2$O$_3$ can be used as the electrolyte at operating temperature of 650 °C. However, δ-Bi$_2$O$_3$ is only stable above 730 °C. When cooled below 730 °C, the appearance of the monoclinic Bi$_2$O$_3$ phase leads to a significant decrease in conductivity, which has limited the application of δ-Bi$_2$O$_3$ to the temperature range of 730 °C–824 °C [14].

Over the past few decades, many studies have investigated the synthesis of single-dopant Bi$_2$O$_3$–based electrolytes with high ionic conductivity, this is because the high-temperature phase of δ-Bi$_2$O$_3$ can be stabilized at room temperature by doping with oxides [15]. Dapčević et al [4] found that Bi$_{1.68}$Tm$_{0.4}$O$_3$ exhibited a high conductivity of 0.117 S cm$^{-1}$ at 550 °C. Vasundhara et al [16] increased the conductivity value to 1.8 × 10$^{-3}$ (Ω
cm$^{-1}$ at 627 °C for the Yb$_{0.56}$Bi$_{0.50}$O$_{1.50}$ binary system [16, 17]. Verkerk et al measured the conductivity of 20ESB to be 0.37 S cm$^{-1}$ at 700 °C, which was the highest reported conductivity among all the single dopant systems [18].

Recently, it was reported that the ionic conductivity of co-doped Bi$_2$O$_3$-based electrolytes was higher than that of the single-dopant components [19–28]. Meng et al showed that compared with single doping, the co-doped samples could stabilize the cubic phase of Bi$_2$O$_3$ under a lower total doping concentration [29]. Because single doping requires a large amount of dopant, it causes greater lattice distortion and results in a low conductivity. The doping of rare earth elements (e.g. Tm, Dy, Nd and Sm) [13, 28, 30, 31] and transition metal oxides (e.g. V, HI, Ti and W) [14, 32–34] in bismuth oxide-based electrolytes can improve their conductivity. Jung et al [33] found that the Dy and W co-doping clearly enhanced the conductivity. In this work, Lu and Ti were co-doped because Lu is a rare earth element and the ionic radius of Lu$^{3+}$ is close to that of Dy$^{3+}$, while the ionic radius of Ti$^{4+}$ is close to that of W$^{6+}$. Yun et al found that the conductivity of Hf$^{4+}$ doped in ESB was very high [34]. Ti and Hf also have a +4 valence, and co-doping with Lu$^{3+}$ and Ti$^{4+}$ may be effective.

To our knowledge, the effect of co-doping Bi$_2$O$_3$ with Lu$^{3+}$ and Ti$^{4+}$ on the oxide ionic conductivity has not been reported. Here, Lu$_2$O$_3$ and TiO$_2$ were selected as co-dopants to stabilize the $\delta$ phase Bi$_2$O$_3$ ceramics, and the sample with the highest conductivity was obtained via a feasible solid-phase method. The morphology, microstructure and electrical properties of the TLSB ceramics were also investigated in detail.

2. Experimental

Bi$_2$O$_3$ (99.9%), Lu$_2$O$_3$ (99.99%) and TiO$_2$ (99.99%) powder (Aladdin Industrial Corporation) were used as raw materials, the Bi$_{2-x}$Lu$_{x}$Ti$_{0.5}$O$_{1.5}$ (where x = 0.04, 0.06, 0.08, 0.10) and Bi$_{1-3y}$Lu$_{2y}$Ti$_{0.5}$ (where x = 0.05, 0.06, 0.07, 0.09, 0.10) samples were synthesized via a solid-state reaction method. The four former compositions, with a fixed 24 mol.% total dopant concentration, are abbreviated by 4T20LSB, 6T18LSB, 8T16LSB and 10T14LSB. The latter five compositions are abbreviated by 5T10LSB, 6T12LSB, 7T14LSB, 9T18LSB and 10T20LSB, respectively. First, the required amount of the oxides was calculated based on the stoichiometry. An electronic balance was used for weighing, the weighed powders were ground for 1 h in an agate mortar mixed with ethanol, and then calcined at 700 °C for 5 h (heating rate of 5 °C min$^{-1}$). The obtained powder was then pressed into pellets with a diameter of 13 mm and thickness of ~1 mm under 200 MPa. The pellets were thereafter sintered at 800 °C for 5 h at a heating rate of 5 °C min$^{-1}$.

The phase composition of the ceramic pellets was characterized at room temperature via x-ray diffraction spectroscopy (XRD) with Cu Kα radiation ($\lambda = 1.5418$ Å). The XRD patterns were recorded in the 2θ range of 20°–80°. The microstructure of the TLSB pellets were determined via scanning electron microscopy (SEM, Quanta FEG 250, FEI Corporate, Hillsboro, OR). The pellets sintered at 800 °C for 5 h were used for electrochemical characterization; for this silver paste was painted on both sides of the TLSB pellets and a silver wire with a diameter of ~0.2 mm was attached tightly to the center. Then, the ceramic pellets were heated at 800 °C for 0.5 h to guarantee good contact between the pellet and silver paste. For the impedance measurements, the electrochemical workstation (CHI660E) was set to the same parameters with E (0 V), amplitude (0.02 V), frequency (0.01 Hz–1 MHz) and quiet time (2s). The testing temperature ranged from 250 °C to 700 °C with steps of 50 °C. The impedance data were analyzed using an equivalent circuit with Z-View software.

3. Results and discussion

3.1. Phase and microstructure of TLSB

The phase composition of the TLSB samples was characterized by XRD. Figure 1(a) reveals the XRD patterns of TLSB pellets with the same 2:1 dopant ratio between Lu and Ti (5T10LSB, 6T12LSB, 7T14LSB, 8T16LSB, 9T18LSB and 10T20LSB) sintered at 800 °C for 5 h. According to Jung et al [34], for DWSB samples, a doping ratio of 2:1 gives optimal for sample performance. Therefore, we studied the samples with a doping ratio of 2:1 in this work. Compared with the standard spectrum in figure 1(a), only the 8T16LSB samples exhibited the pure $\delta$-Bi$_2$O$_3$ phase. For the 5T10LSB, 6T12LSB, and 7T14LSB samples, some weak impurity peaks could be observed. After comparison with the standard spectrum, it was found that the impurity peaks belonged to the Bi$_{1-x}$Ti$_{0.5}$O$_{2-x}$ and Bi$_2$O$_3$ phases. Further, with increasing total doping concentration, the impurities gradually disappeared. As the total concentration was further increased, in the 9T18LSB and 10T20LSB samples, diffraction peaks at 30.06° and 33.10° could be observed, which corresponded with the (1 1 7) and (0 2 0) crystal surfaces, respectively, and these impurity peaks belonged to the Bi$_3$Ti$_2$O$_9$ phase. Next, a series of investigations were carried out with an unchanged total doping concentration of 24 mol.%. Figure 1(b) reveals the XRD patterns of the TLSB pellets (4T20LSB, 6T18LSB, 8T16LSB, and 10T14LSB) sintered at 800 °C for 5 h. According to figure 1(b), all the other samples had a good single-phase cubic fluorite structure, which was consistent with
the diffraction peaks of the Bi$_2$O$_3$ standard spectrum. There were no distinct additional peaks for the second phase and raw materials, and the doped ions gradually entered into the Bi$_2$O$_3$ lattice, forming the Bi$_{0.76}$Lu$_{0.24-x}$Ti$_x$O$_{1.5+\delta}$ solid solution. Figure 2 is the Rietveld pattern of the samples with unchanged total doping concentration, and Table 1 is the cell parameter value obtained according to the Rietveld analysis. With increasing Ti$^{4+}$ doping and decrease of the Lu$^{3+}$ dopant, the characteristic peak shifted slightly to a smaller angle. This was because replacing Lu$^{3+}$ with Ti$^{4+}$ reduced the oxygen vacancies, and resulted in an increase in the unit cell parameters, which is because the presence of oxygen vacancies causes the unit cell to shrink. The small ionic radius of Ti$^{4+}$ that replaced the large ionic radius Lu$^{3+}$ could lead to the decrease in the unit cell parameters. When the Ti doping concentration was less than 8 mol.%, the combination of the two factors finally led to an increase of the unit cell parameters and a decrease in the diffraction angle, as well as a shift of the diffraction peak to a smaller angle. Figure 3 shows SEM micrographs of the TLSB pellets (including 4T20LSB, 6T18LSB, 8T16LSB, and 10T14LSB) sintered at 800 °C for 5 h. And we calculated the porosity of the samples carefully, which was 5.3%, 5.1%, 4.8%, 4.9%, respectively. The results showed that the porosity of all samples were relatively small, and the porosity first decreased and then increased. According to the porosity, we could get the relative density, all the samples were relatively dense, and the porosity had no effect on the conductivity.

3.2. Electrical performance

The electrical conductivity of the TLSB pellets was investigated over the temperature range of 250 °C to 750 °C via electrochemical impedance spectroscopy (EIS). Figure 4 showed the AC impedance spectra of all studied samples at 250 °C. Figure 4(a) showed the TLSB ceramics with the same 2:1 dopant ratio of Lu and Ti. Figure 4(b) showed the TLSB ceramics with an unchanged total doping concentration of 24 mol.%. Figure 4 consists of a
single semicircle and an arc, with the former arising from the grain and the latter belongs to the electrode contribution; this is because the capacitance interval of the semi-circular arc is $10^{-10}$ F, while that of the arc is $10^{-6}$ F. It could be deduced that the resistance of the ceramic was mainly determined by the grain, and its conductivity could be calculated from the grain resistance value. The high-frequency semicircle caused by grain resistance could no longer be distinguished with increasing temperature. As shown in figure 4, the impedance of 8T16LSB sample was minimum, which was consistent with the subsequent conductivity results. The grain resistance value was obtained from the high-frequency intercept, and the total conductivity was calculated from the measured grain resistance and electrode area.

The Arrhenius plot of the TLSB ceramics with the same 2:1 dopant ratio between Lu and Ti (5T10LSB, 6T12LSB, 7T14LSB, 8T16LSB, 9T18LSB, and 10T20LSB) is shown in figure 5(a). It can be seen that as the total doping concentration increases, the conductance first increased and then decreased, and the conductivity of 8T16LSB reached a maximum. Then, the doping ratio was changed while maintaining a constant total doping concentration of 24 mol.%. Figure 5(b) shows the Arrhenius plot of the TLSB (including 4T20LSB, 6T18LSB, 8T16LSB, and 10T14LSB) ceramics and the conductivity of the 20ESB from literature for comparison [35] is also indicated. It can be seen that the conductivity first increased and then decreased with increasing Lu to Ti doping concentration. When the Ti and Lu doping concentration was 8 mol.% and 16 mol.% respectively, the highest conductivity was 0.56 S cm$^{-1}$ at 700°C for the 8T16LSB specimen. When the Ti doping concentration was less than 8 mol.%, the conductivity increased continuously. The first contribution is because Ti replaced Lu, and Ti

![Figure 2. The Rietveld patterns of TLSB pellets (a) 4T20LSB (b) 6T18LSB (c) 8T16LSB and (d) 10T14LSB.](image)

| Sample   | a(Å)   | b(Å)   | c(Å)   | V(Å$^3$) | $R_{wp}$(%) | $\chi^2$ |
|----------|--------|--------|--------|----------|------------|----------|
| 4T20LSB  | 5.4691 | 5.4691 | 5.4691 | 163.59   | 5.67       | 2.875    |
| 6T18LSB  | 5.4780 | 5.4780 | 5.4780 | 164.39   | 6.09       | 2.921    |
| 8T16LSB  | 5.4821 | 5.4821 | 5.4821 | 164.76   | 4.96       | 2.378    |
| 10T14LSB | 5.4802 | 5.4802 | 5.4802 | 164.59   | 5.14       | 2.656    |
is more electronegative than Lu; the introduction of a large number of more electronegative ions weakened the chemical bond between the cation and oxygen, hence reducing the activation energy and increasing the conductivity. Second, after Ti$^{4+}$ replaced Lu$^{3+}$, the oxygen vacancy concentration decreased and the cell parameters increased, which resulted in reduced lattice distortion and activation energy, resulting increased the electrical conductivity. These two synergistic effects on the TLSB ceramics acted to promote their conductivity. When the doping concentration reached 10 mol.%, the conductivity decreased instead. The reason for this is that the oxygen vacancy concentration as well as the preexponential factor further decreased with increasing Ti

Figure 3. SEM micrographs of TLSB (including 4T20LSB, 6T18LSB, 8T16LSB, and 10T14LSB) (a)$x = 0.04$ (b)$x = 0.06$ (c)$x = 0.08$ (d)$x = 0.10$ pellets sintered at 800 °C for 5 h.

Figure 4. The impedance spectra of all studied samples were measured in at 250 °C (a) the ceramics with the same 2:1 dopant ratio of Lu and Ti (b) the ceramics with an unchanged total doping concentration of 24 mol.%. 

(a)

(b)
The Nyquist plots of 8T16LSB exposed to different duration periods are shown in Figure 6. The conductivity of the sample decreased slightly after 30h to $\sim 0.19 \text{ S cm}^{-1}$ at 600 $^\circ$C, which was still higher than that of 1HESB at 600 $^\circ$C ($\sim 0.14 \text{ S cm}^{-1}$) reported in the literature [33].

From Figure 5, it can be seen that the conductivity of the 8T16LSB sample in this paper reached the highest value of 0.56 S cm$^{-1}$ at 700 $^\circ$C. This demonstrated that the conductivity after co-doping was higher than that realized with single-dopant, which includes 20ESB with the highest conductivity of 0.37 S cm$^{-1}$ at 700 $^\circ$C. In Figure 5, it can be seen that the conductivity of the 8T16LSB sample in this paper reached the highest value of 0.56 S cm$^{-1}$ at 700 $^\circ$C. This demonstrated that the conductivity after co-doping was higher than that realized with single-dopant, which includes 20ESB with the highest conductivity of 0.37 S cm$^{-1}$ at 700 $^\circ$C. As reported in the literature. Therefore, the 8T16LSB ceramics are suitable for use as an oxygen ionic conductor in SOFCs.

4. Conclusions

In summary, a new oxygen ion electrolyte material TLSB was synthesized via a solid-state reaction method. The XRD results showed that after sintering at 800 $^\circ$C for 5 h, all the samples with a fixed total dopant concentration of 24 mol.% exhibited a single cubic fluorite structure, and there were no impurity and raw material phases. This indicated that the doped ions entered the Bi$_2$O$_3$ lattice and formed a stable solid solution. According to the SEM results, all the TLSB ceramics (including 4T20LSB, 6T18LSB, 8T16LSB, and 10T14LSB) were relatively dense.

The electrochemical characterization revealed that the 8T16LSB ceramic reached the highest conductivity of 0.56 S cm$^{-1}$ at 700 $^\circ$C. In the current study, the conductivity after co-doping was observed to be higher than that realized with any single-dopant, including for 20ESB with the highest conductivity of 0.37 S cm$^{-1}$ at 700 $^\circ$C. In
conclusion, this work demonstrated that the 8T16LSB sample was a promising electrolyte material with high oxygen ionic conductivity for practical application.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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Figure 6. The Nyquist plots of 8T16LSB exposed to different duration periods.
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