Highly oxygen-permeable and CO2-stable Ce0.8Sm0.2O2 − δ−SrCo0.9Nb0.1O3 − δ dual-phase membrane for oxygen separation

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Abstract

Dual-phase composite oxide 60 wt% Ce0.8Sm0.2O2 − δ−SrCo0.9Nb0.1O3 − δ (60SDC-40SCN) which exhibits high oxygen permeability and good CO2 tolerance was developed. X-ray diffraction (XRD) patterns and dense surface topography observed by scanning electron microscopy (SEM) revealed a good compatibility of the two oxides. A high oxygen permeation flux of 1.54 mL min−1 cm−2 through the as-prepared dual-phase membrane (0.8 mm in thickness) was obtained under the gradient of air/He at 1223 K. In situ high-temperature X-ray diffraction demonstrated that SDC and SCN in 60SDC-40SCN membrane could retain their original phase structure from room temperature to 1223 K in CO2-containing atmosphere. The oxygen permeation fluxes of 60SDC-40SCN membrane showed a good reversibility when switching the sweep gas between CO2 and He. Comparing with single-phase SCN membrane, oxygen permeation flux of dual-phase 60SDC-40SCN membrane was more than twice when pure CO2 acted as the sweep gas, and the oxygen permeation flux could remain stable for 120 h. All the experimental results imply that dual-phase 60SDC-40SCN membrane has a great potential in oxy-fuel combustion process.

1. Introduction

With greenhouse gases build-up in the atmosphere, global warming has become a public concern. Among all the sources of greenhouse gases emission, combustion of fossil fuel or coal in power plants is a primary polluter of carbon dioxide which needs to be addressed urgently. As one of the most promising CO2 capture technologies, oxy-fuel process has been carried out to promote and optimize the combustion of fossil fuel and coal [1–3]. A typical oxy-fuel combustion process uses pure oxygen instead of air for fuel combustion, resulting in a concentrated CO2 gas stream for the subsequently high-efficiency CO2 capture to realize emission reduction of greenhouse gases [4,5]. Nevertheless, oxygen supply come from conventional air separation technologies on the basis of cryogenic distillation and pressure swing adsorption require large scale energetic and economic costs. Alternatively, mixed ionic electronic conducting (MIEC) oxygen permeation membrane process, which can be integrated with the combustion process providing pure oxygen in a lower implementation costs, may have great potential for oxygen separation [6,7]. Furthermore, because MIEC oxygen permeation membranes allow 100% oxygen selectivity [8], the emissions of NOx can also be reduced [9] and carbon capture and sequestration process can be implemented during combustion [10].

Over the past few decades, a large amount of research activities have been focused on MIEC oxides development, and a series of state-of-the-art materials with good oxygen permeation performance were developed [11]. Among them, Co-containing perovskite-type oxides, such as Ba0.5Sr0.5Co0.8Fe0.2O3−δ (BSCF) [12], SrCo0.8Fe0.2O3−δ [13], which exhibit very high oxygen permeability, were considered as the most promising membrane materials for industrialization. Unfortunately, their poor stability in CO2 or SO2 atmosphere limits their practical applications in oxygen permeation membranes. This attributes to the alkaline earth metal elements (such as Ba2+ and Sr2+ which have large ionic radii) on A site show susceptible to the acidic gas and easily occur reactions forming carbonates and sulfates [14,15]. In addition, the variation in the valence of cobalt makes it behave imperfectly in terms of structure stability [16,17]. Alternatively, dual-phase composite oxides which allow ions and electrons transport in different phases show superiority in stable oxygen permeability under CO2 atmosphere [18], such as NiFe2O4−Ce0.1TD0.9O2−δ [9], Nd0.5Sr0.5FeO3−δ−Ce0.1Nd0.9O2−δ [19], Ce0.9Gd0.1O2−δ−NiFe2O4 [20], Ce0.8Gd0.2O2−δ−La0.7Sr0.3MnO3−δ [21], Ce0.8Sm0.2O2−δ−La0.8Sr0.2CrO3−δ [22] and Zr0.8Y0.16O1.92−La0.8Sr0.2CrO3−δ [23].

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However, on account of the inefficient electronic and oxygen ions transport [7,24], the poor oxygen permeability of most dual-phase membranes cannot meet the industrial requirements. Because single-phase mixed conducting oxides with highly oxygen-permeable, such as Co-containing oxides, still play an irreplaceable role in future practical application, how to improve stability of these materials is an important issue demanding prompt solution. According to the design strategy of dual-phase oxygen permeation membrane mentioned above, we report a method that doping fluoride-type oxides into the Co-containing composites to achieve highly oxygen permeable and CO₂ stable dual-phase membrane materials. Here we select Ce₀.₈Sm₀.₂O₁₋₇ (SCN) which has good fast ions conductivity and good performance in chemical durability as oxygen ion transport materials. Perovskite oxide SrCo₀.₉Nb₀.₁O₃₋₆ (SDC) which has good structure stability [13] and high oxygen permeability [25] act as the oxygen ion and electronic conductors. In this work, phase structures and surface morphology of the as-prepared dual-phase membranes were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM) respectively. To test the oxygen permeability of the dual-phase membranes, the oxygen permeation fluxes as functions of SDC/SCN mass ratios and membrane thickness were investigated. In order to examine the CO₂ tolerance of the dual-phase membrane, the oxygen permeability, the phase structure change and chemical stability were investigated systematically under different CO₂ atmospheres.

2. Experiment

2.1. Preparation of SDC and SCN powders

The powder of SDC was synthesized via a combined EDTA-citrate complexing sol-gel method [26]. Briefly, the required amounts of analytical grades metal nitrates of Sm (NO₃)₃ and Ce (NO₃)₃ were dissolved in aqueous solution, followed by the addition of EDTA, the molar ratio of citric acid:EDTA:total metal ions was 1:5:1. The pH value of the solution was adjusted in the range of 6–8 by the addition of ammonium hydroxide. Under the condition of 363 K heating and stirring, the gel was obtained with the evaporating water. The gel was pre-fired in the air at 523 K to remove organic compounds and further calcined at 1073 K for 5 h under air atmosphere to obtain desired structural composition.

SrCo₀.₉Nb₀.₁O₃₋₆ powder was fabricated by the conventional sol-gel reaction techniques. The required stoichiometric amounts of SrCO₃, CoO and Nb₂O₅ were mixed in the ethanol solution and then ball milled for 24 h. After the solvent evaporated, the mixed oxides were calcined in muffle furnace at 1223 K for 5 h with heating and cooling rates of 5 K min⁻¹. The sintered SCN powder was ground and sieved (300 meshes) to prepare the dual-phase composite oxides and membranes.

The dual-phase composite oxides 50 wt% SDC-50 wt% SCN, 60 wt% SDC-40 wt% SCN, 75 wt% SDC-25 wt% SCN were mixed in a agate jar and ball milled for 10 h to prepare homogeneous dual-phase composite oxides.

2.2. Membrane preparation and characterization

The dual-phase composites were pressed into disks under a pressure of ~200 MPa. The green disks were sintered at 1373 K for 5 h. The crystal phase structures of the SCN, SDC and different compositions of dual-phase membranes were characterized by X-ray diffraction (XRD, Bruker, model D8 Advance) with Cu Kα radiation. The variation of crystal structures with temperature were characterized by in situ XRD equipped with a high temperature attachment (in situ HTXRD, Philips, XPert Pro). The samples were tested in air and 50 vol% N₂/50 vol% CO₂ atmosphere from 298 K to 1273 K with a heating rate of 5 K min⁻¹ and each temperature point was held for 60 min for thermal equilibrium before diffraction data collection. All of the diffraction patterns were collected by step scanning at an increment of 0.05° in the range 20° ≤ 2θ ≤ 80°. The morphology and micro-structure of the single and dual-phase membranes were examined by environmental scanning electron microscopy (SEM, Hitachi S-4800, Japan). A gas-tight test was conducted on the sintered asymmetric membrane using nitrogen at room temperature.

2.3. Oxygen permeation flux measurement

Oxygen permeation fluxes through the single-phase and dual-phase membranes were measured by high-temperature oxygen permeation device which can be seen in our previous work [27]. A disk membrane with effective diameter of 7.5 mm was sealed on the end of an alumina tubes with silver adhesives. Temperatures of the oxygen permeation measurement device were monitored by a programmable temperature controller (AI-708PA, Yudian, China). Flow rates of the feed and sweep gas were controlled by mass flow controllers (D07-19B, Sevenstar, China). The gaseous mixture were analyzed by an On-Line gas chromatograph (GC-8A, Shimadzu, Japan), which equipped with a 2 m 5 A molecular sieve with helium (He) as the carrier gas. The leakage of the oxygen due to the defective sealing at high temperatures was <5% of the total oxygen permeation flux during all the experiments. The computational methods of oxygen permeation flux have also illustrated in our previous works [28].

3. Results and discussion

3.1. Phase and microstructure

The XRD patterns of dual-phase membranes sintered at 1373 K for 5 h are shown in the Fig. 1. As references, single-phase SDC and SCN are also included. For SDC pattern, the diffraction peaks at the respective 2-theta angles of 28.42° (111), 32.96° (200), 47.28° (220), 56.12° (311), 58.86° (222) and 76.42° (313) matches the fluorite structure. And for SCN, the 2-theta angles of 32.72° (110), 40.5° (111), 47.06° (200), 58.42° (211), 68.44° (220) and 77.86° (310) corresponding to the perovskite structure. No additional phase resulting from undesirable reaction can be found from the diffraction peaks of the 60SDC-40SCN dual-phase at the room temperature, suggesting that the sintered membrane consists of only SDC and SCN phases. The patterns of the 50SDC-50SCN and 75SDC-25SCN are also depicted, same as the 60SDC-40SCN, no significant formation of impure phases can be found, revealing the stable coexistence of SDC and SCN in dual-phase membrane.

Fig. 1. XRD patterns of the SCN, SDC and SDC-SCN membranes. 50SDC-50SCN, 60SDC-40SCN and 75SDC-25SCN denote 50 wt% Ce₀.₈Sm₀.₂O₁₋₇, 60 wt% Ce₀.₈Sm₀.₂O₁₋₇ and 75 wt% Ce₀.₈Sm₀.₂O₁₋₇ respectively.
Fig. 2 presents the SEM micrographs of the single-phase SCN and dual-phase SDC-SCN membranes sintered at 1373 K for 5 h made by the powder mixed method. The SCN membrane (Fig. 2a) presents dense and crack-free surface structure, and the size of the polyhedral independent particles are in the range of 3–4 μm. Fig. 2b shows the surface topography of the 50SDC–50SCN membrane. As shown, particles with size in the range of 300 nm–1 μm are represent for fluorite SDC (light-colored grains), and perovskite SCN (dark-colored grains) mostly scattered in the range of 500 nm–1 μm. The crack-free surface structure and clear grain-boundaries suggest a good chemical compatibility of the two oxides. It is worth noting that the particle size of SCN in dual-phase membrane is smaller than that in the SCN membrane, which attributes to the constraint effect of the SDC oxides on SCN composites when sintered together. As we know, for most MIEC oxides (such as Ba0.5Sr0.5Co0.8Fe0.2O3−δ), grain growth will decrease the number of grain boundaries and has positive effect on oxygen permeation process. On the contrary, for most dual-phase membrane, because the electronic and ionic transport by different paths, grain growth will break the continuity of electronic and ionic conductors. Therefore, the result of grain size shrinkage will increases the areas of phase boundary and has a positive effect on oxygen permeability [29].

Fig. 3 shows the temperature dependence of oxygen permeation fluxes of SCN and three dual-phase membranes. As expected, the oxygen permeation fluxes through all the membranes increase with the rising temperature, which attributed to the promoted surface exchange kinetics and oxygen ions diffusion in higher temperatures. Among the three dual-phase membranes, 60SDC–40SCN shows the best oxygen permeability. At 1223 K, oxygen permeation flux through the 60SDC–40SCN membrane is about 1.54 mL min⁻¹ cm⁻², which is higher than the flux of 50SDC–50SCN membrane in the same condition. It is because the incremental SDC which act as the ionic conductor can accelerate the transportation of oxygen ions through the membrane. However, the oxygen permeation flux of 75SDC–25SCN is not accord with the above conclusion, at 1223 K the oxygen flux of the 75SDC–25SCN is only 0.46 mL min⁻¹ cm⁻². This attributes to the discontinuous ionic or electronic transport paths formed by excess ionic conductors prevent the process of oxygen permeation [30].

As we know, oxygen transport through the dense oxygen permeation membrane is controlled by two factors: the surface-exchange kinetics and the bulk diffusion rate of electrons and ions [31,32]. In order to confirm the effects of the two processes on oxygen permeation flux of dual-phase membrane, the relationship of 60SDC–40SCN membrane thickness with the oxygen permeation flux were investigated. According to Fig. 4a, increased oxygen permeation fluxes are observed
when decrease the thickness of the membrane. For example, at 1223 K, the permeation fluxes are 1.54, 1.29 and 1.09 mL min\(^{-1}\) cm\(^{-2}\) for membranes with the thickness of 0.8, 1.0, and 1.2 mm, respectively. Further calculation demonstrates that the value \(J_{\text{O}_2}L\) for the three membranes almost remain constant as the function of thickness. In details, the \(J_{\text{O}_2}L\) value of the 0.8, 1.0 and 1.2 mm thick membrane are 1.23, 1.29, 1.31 at 1223 K, which means that the oxygen transport is mainly controlled by the oxygen bulk diffusion when the thickness of membrane beyond 0.8 mm [33]. However, it is worth noting that the increase of oxygen permeation fluxes were not obvious when the thickness of membrane decrease <0.8 mm. Fig. 4b shows the oxygen permeation fluxes against the reciprocal of thickness from 1073 to 1223 K. It can be seen obviously that as the thickness decrease to 0.6 mm the value of oxygen flux deviates from the linear trend, suggesting that the surface-exchange kinetics is not negligible in the oxygen permeation process on this occasion [34,35]. Therefore, further decrease the thickness of membrane won’t increase the oxygen permeation flux of dual-phase membrane.

3.3. \(\text{CO}_2\)-tolerant measurement

Fig. 5 shows the oxygen permeation fluxes of SCN and 60SDC-40SCN dual-phase membranes as a function of time in the condition of periodically changing the sweep gas from He to \(\text{CO}_2\) at 1223 K. As can be seen in the Fig. 5a, the oxygen permeation flux of the SCN is about 0.23 mL min\(^{-1}\) cm\(^{-2}\) when \(\text{CO}_2\) acts as the sweep gas at 1223 K. Compared with the oxygen flux of SCN membrane swept by He (about 2.61 mL min\(^{-1}\) cm\(^{-2}\)), the oxygen permeation flux through SCN membrane dropped by 90%. Moreover, when the sweep gas change from \(\text{CO}_2\) to He, the oxygen flux through SCN membrane increased by 110%.
to He again, the oxygen permeation flux through the SCN membrane cannot recover the original value immediately, which may attribute to the retarded oxygen exchange rates caused by the adsorption of CO2 on the membrane surface [4,15,20]. Fig. 5b displays the oxygen permeation performance of 60SDC-40SCN membrane in the same condition as above. The oxygen permeation flux of 60SDC-40SCN membrane swept by CO2 is 0.52 mL min\(^{-1}\) cm\(^{-2}\) at 1223 K. Following the sweep gas shift from CO2 to He again, the oxygen permeation flux recover the original value (1.53 mL min\(^{-1}\) cm\(^{-2}\)). Therefore, compared with the single-phase SCN membrane, dual-phase membrane shows a better oxygen permeability and stability in CO2 atmosphere.

The influence of different concentration of CO2 in the sweep side on oxygen permeation fluxes of SCN and dual-phase SDC-SCN membranes were also investigated. As shown in the Fig. 6a, when CO2 concentration increase from 0 to 10%, oxygen permeation flux of SCN membrane varies from nearly 2.6 mL min\(^{-1}\) cm\(^{-2}\) to 2.2 mL min\(^{-1}\) cm\(^{-2}\); and for the 60SDC-40SCN membrane the oxygen permeation flux decrease from 1.53 to 1.36 mL min\(^{-1}\) cm\(^{-2}\) at 1223 K; for the 75SDC-25SCN membrane the oxygen permeation flux has little change. When the concentration of CO2 increase to 50%, the SCN and 50SDC-50SCN membranes exhibit about 85% and 60% oxygen permeation fluxes decrease respectively, and the 60SDC-40SCN dual-phase membrane displays a less 50% drop. However, for the 75SDC-25SCN membrane when the concentration of CO2 increases from 0 to 100%, only 21% oxygen permeation flux decline is found. The histograms shown in Fig. 6b describe the change of oxygen permeation fluxes of the SCN and three dual-phase membranes treated by pure CO2 in the sweep side. It can be seen obviously that 60SDC-40SCN membrane possesses the highest oxygen flux (twice the value of single-phase SCN membrane), and 75SDC-25SCN membrane exhibits the best CO2-tolerance. These findings demonstrate the improved CO2 tolerant of dual-phase membrane made by the introduction of SDC into SCN oxides.

Fig. 7a shows the in-situ XRD patterns of sintered SCN powders in air atmosphere. During the temperatures increasing from room temperature to 1273 K, no additional phase except perovskite-type SCN was observed. Additionally, the high temperature phase stability of SCN in an atmosphere of 50 vol% CO2-50 vol% N2 was also studied under the same test conditions. As shown in Fig. 7b, the phase structure of SCN composite is essentially constant before 1073 K. As temperature increasing successively, impure peaks appear and the major peaks of the SCN become wide and weak, suggesting the instable phases structure of SCN, that’s why the oxygen permeation flux of SCN membrane decline sharply in the atmosphere of CO2. However, compared with the single SCN material, SDC doped SCN membrane exhibits a good phase stability in the atmosphere of CO2. As shown in the Fig. 8b, when treated by the 50 vol% CO2 and 50 vol% N2, no obvious miscellaneous phase formation can be detected from the XRD patterns of 60SDC-40SCN composite, indicating the stability in a CO2-containing atmosphere.

Fig. 9 shows the time dependence of oxygen permeation fluxes of 60SDC-40SCN membrane swept by pure CO2 at 1223 K. As shown, the oxygen permeation fluxes through the dual-phase membrane vary slightly in the range 0.49 to 0.53 mL min\(^{-1}\) cm\(^{-2}\), and no apparent decline is observed. After the long-term stability measurement with 60 mL min\(^{-1}\) pure CO2 as the sweep gas, the feed side and permeation side of the dual-phase membrane were analyzed by XRD. As shown in the Fig. 10, both SDC and SCN remain their own crystal structure, and phase structure of the dual-phase membrane in feed side almost constant. In the sweep side, some new diffraction peaks belong to Ag (which acts as the sealed material) can be found. Additionally, the diffraction peaks of SCN of the dual-phase membrane in the sweep side become weak, and accompanied by the generation of trace amounts impure phases, this change possibly come from the diffusion of alkaline earth metals forming carbonates in the surfaces of membrane [36].

Fig. 11 shows SEM images of the fresh and used 60SDC-40SCN membranes. The cross-section of fresh membranes has a typical intercrystalline discontinuity microstructure with grain boundary clearly visible. The spent membranes shown in Fig. 11b exhibit the similar cross-section morphology to the fresh membrane. Surface microstructure of the membranous feed side (Fig. 11c) remains dense and crack-free. Compared with the fresh membrane surface microstructure in Fig. 3, the surface of the feed side becomes rough after permeation measurement. In case of the sweep side, as shown in Fig. 11d, significant etching feature can be found. It is principally related to the formation of SrCO3 during the dwelling time of 120 h. However, from the SEM image of the cross section in Fig. 11b, we can confirm that the etching feature only forming in the surface layer [7,36,37].

Table 1 lists the oxygen permeation fluxes of several types of dual-phase membranes. As shown, the dual-phase materials which only use pure electronic conductor, like Pd, Fe2O3 and NiFe2O4, act as the electronic transporting phase exhibit low oxygen permeable because the blockade of ionic transportation [41,42]. Then, mixed conductors were proposed to be substituted for pure electronic conductor to overcome the blockade. Because the mixed conductor with higher ionic conductivity as the electronic conductor can assist the oxygen ionic transportation, doping Co-containing composites into the fluorite-type oxides is an effective method to achieve highly oxygen permeable and CO2 stable dual-phase membrane materials. By using this type of dual-phase oxygen permeation membrane, oxygen can be separated from air by using the exhaust gas CO2 as sweep gas, which can reduce costs and save energy compared with the conventional cryogenic process [3].

4. Conclusion

Dual-phase composites oxygen permeation membranes based on Ce0.8Sm0.2O2−δ−δ SrCo0.9Nb0.1O3−δ were successfully developed. The oxygen permeability of different SDC proportions dual-phase composite membrane was investigated. And the 60SDC-40SCN membrane exhibited the best oxygen permeability and relatively better CO2 tolerance. In the condition of helium sweeping, the oxygen permeation flux of 60SDC-40SCN membrane (0.8 mm in thickness) reached as high as
1.54 mL min$^{-1}$ cm$^{-2}$ at 1223 K. \textit{In situ} high-temperature X-ray diffraction evidenced that SDC and SCN in 60SDC-40SCN membrane could remain their original structure from room temperature to 1223 K in air and 50 vol\% CO$_2$/50 vol\% N$_2$, which demonstrated the good chemical stability of 60SDC-40SCN dual-phase membrane in CO$_2$-containing atmosphere. During the 120 h oxygen permeation measurement at 1223 K with pure CO$_2$ as the sweep gas, 60SDC-40SCN (0.8 mm in thickness) membrane possessed a stably high oxygen permeation fluxes of 0.52 mL min$^{-1}$ cm$^{-2}$. All the results demonstrate that the 60SDC-40SCN membrane exhibits high oxygen permeability and good stability in CO$_2$ atmosphere, which indicates a great potential application in oxy-fuel combustion and CO$_2$ capture and storage.

Fig. 8. \textit{In situ} XRD patterns of 60SDC-40SCN dual-phase composite for increasing temperatures under (a) air and (b) 50 vol\% CO$_2$ and 50 vol\% N$_2$. Conditions: heating rate = 5 K min$^{-1}$, equilibration time: 30 min for recording the XRD data at each temperature.

Fig. 9. Oxygen permeation fluxes through 60SDC-40SCN membranes as a function of time at 1223 K. Conditions: \( F_{\text{Air}} = 120 \text{ mL min}^{-1}, F_{\text{CO}_2} = 60 \text{ mL min}^{-1} \), thickness = 0.8 mm.

Fig. 10. XRD patterns of the spent 60SDC-40SCN membranes after treatment in pure CO$_2$ atmosphere for 120 h.
Acknowledgements

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Fig. 11. SEM micrographs of the fresh and spent 60SDC–40SCN membrane. (a) Cross-section of fresh membrane; (b) cross-section of the spent membrane. (c) Feed side the spent membrane. (d) Permeation side of the spent membrane.

Table 1

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<th>Membrane composition</th>
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<th>J0 (mL min−1 cm−2) Air/CO2</th>
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