Electronic conductivity, oxygen permeability and thermal expansion of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_3$-

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Abstract

The maximum solubility of aluminum cations in the perovskite lattice of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_3$ is approximately 15%. The incorporation of $\text{Al}^{3+}$ increases oxygen ionic transport due to increasing oxygen nonstoichiometry, and decreases the tetragonal unit cell volume and thermal expansion at temperatures above 600 °C. The total conductivity of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_3$ ($x=0-0.2$), predominantly electronic, decreases with aluminum additions and has an activation energy of 10.2–10.9 kJ/mol at 350–850 °C. Analysis of the electronic conduction and Seebeck coefficient of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_0\text{O}_3$, measured in the oxygen partial pressure range from $10^{-4}$ to 5 atm at 700–950 °C, revealed trends characteristic of broad-band semiconductors, such as temperature-independent mobility. The temperature dependence of the charge carrier concentration is weak, but exhibits a tendency to thermal excitation, whilst oxygen losses from the lattice have an opposite effect. The role of the latter factor becomes significant at temperatures above 800 °C and on reducing $p(\text{O}_2)$ below $10^{-4}$ to $10^{-2}$ atm. The oxygen permeability of dense $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_3$ membranes, limited by both bulk ionic conduction and surface exchange, is substantially higher than that of $\text{(La, Sr)MnO}_3$-based materials used for solid oxide fuel cell cathodes. The average thermal expansion coefficients of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_3$ ceramics in air are $(10.8-11.8) \times 10^{-6} \text{K}^{-1}$.

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1. Introduction

Electrical power generation in solid oxide fuel cells (SOFCs) provides substantial advantages with respect to traditional energy conversion systems including high efficiency, reliability, modularity, fuel adaptability, low noise and low environmental impact. One of the most important challenges in SOFC developments relates to decreasing operation temperatures, which improves long-term performance due to suppressing interaction between different ceramic layers, enhances microstructural stability of electrodes, increases thermodynamic efficiency and enables to use cheaper materials for stack components and SOFC interconnects. As the activation energy of the electrolyte polarization is higher than that of the ionic transport, the performance of intermediate-temperature (IT) SOFCs is typically limited by the electrode overpotentials. Conventional cathodes based on perovskite-type $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ have a poor oxygen ionic conductivity and insufficient electrocatalytical activity at intermediate temperatures.

Recently, a new group of manganites, $\text{Sr}_{1-x}\text{Ce}_x\text{MnO}_3$, was suggested as promising cathode materials for SOFCs. $\text{SrMnO}_3$ has a perovskite-type lattice at temperatures above 1400 °C, but undergoes phase transition to hexagonal 4L structure on cooling below 1035 °C. Partial substitution of strontium with Ce cations stabilizes the perovskite phase down to room temperature and increases the total conductivity by two to three orders of magnitude at 600–1000 °C. The use of $\text{Sr}_{1-x}\text{Ce}_x\text{MnO}_3$ may be advantageous due to higher level of the oxygen ionic conductivity with respect to $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$. Although
this level is still lower than for other promising SOFC cathodes, such as La1−xSrxFe1−yCoyO3−δ, the ionic transport of (Sr, Ce)MnO3-based materials can be enhanced by partial substitution of manganese. In particular, as oxygen diffusion in perovskite-type phases occurs via the vacancy mechanism, an enhancement in the ionic conduction may be achieved incorporating acceptor-type cations (e.g. Al3+), thus increasing oxygen vacancy concentration. Doping with aluminum may also suppress the interaction between Sr1−xCexMnO3−δ and stabilized zirconia electrolytes. Moreover, heating of the cathode materials is often accompanied with oxygen losses from the lattice, resulting in so-called chemical contribution to the thermal expansion. This may cause interface instability. The substitution of manganese with cations having a constant oxidation state is expected to suppress oxygen stoichiometry variations, as for other transition metal-containing perovskites.

The present work was focused on the study of Sr1−xCexMn1−xAlxO3−δ system, with particular attention to the properties important for high-temperature electrochemical applications, namely thermal expansion, oxygen transport and electronic conduction.

2. Experimental

The powders of Sr0.7Ce0.3Mn1−xAlxO3−δ (x = 0–0.3) were prepared via the standard solid-state synthesis route from high-purity Sr(NO3)2, Ce(NO3)3·6H2O, Mn2O3 and Al(OH)3. The stoichiometric amounts of reagents were initially dissolved in dilute nitric acid, followed by drying of the solution and thermal decomposition of the nitrate mixture at 400 °C. The solid-state reactions were conducted at 900–1350 °C in air with multiple intermediate grinding steps. After ball milling, disk-shaped ceramic samples were pressed at 150–200 MPa and then sintered at temperatures from 1320 °C (x = 0) up to 1450–1500 °C (x = 0.1–0.3). The density of sintered ceramics was higher than 93% of their theoretical density calculated from X-ray diffraction (XRD) data; the samples for electrical and oxygen permeation measurements were additionally tested for gas-tightness. General characterization of the ceramic materials included scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS), dilatometry, determination of Seebeck coefficient and total conductivity (four-probe dc) as function of the oxygen partial pressure, and steady-state oxygen permeability measurements. Description of the experimental techniques and equipment used for characterization can be found elsewhere (Refs. 19–23 and references cited therein). The Seebeck coefficient and total conductivity were studied in the p(O2) range 10–19 to 0.5 atm, as described in Refs. 22, 23. The steady-state oxygen permeability was measured at 900–975 °C; the feed-side oxygen partial pressure (p2) for all oxygen permeation measurements was 0.21 atm.

3. Results and discussion

3.1. Phase composition, microstructure and thermal expansion

XRD studies of Sr0.7Ce0.3Mn1−xAlxO3−δ ceram showed the formation of single-phase perovskite for x = 0–0.1 (Fig. 1). Further doping leads to segregation of impurity phases, including CeO2 and SrMn1O6; the intensity of impurity peaks increases with increasing aluminum content. This indicates that the maximum solubility of Al3+ cation in the B-sublattice of Sr0.7Ce0.3MnO3−δ is close to 15%. Crystal structure of Sr0.7Ce0.3Mn1−xAlxO3−δ was identified as tetragonally-distorted perovskite, in agreement with on undoped Sr0.7Ce0.3MnO3−δ. Therefore, although substitution of manganese with aluminum increases the oxygen vacancy concentration as evidenced by the oxygenation data presented further, no destabilization of the perovskite lattice occurs. The unit cell parameters decrease increasing x (Table 1) as the ionic radius of Al3+ is smaller than that of manganese cations. Fig. 1 also shows that structural collapse occurs under strongly reducing conditions; cause reduction of Mn2+ to lower valence yields significant size changes; the approximate phase stability limit discussed further.

SEM/EDS inspection of Sr0.7Ce0.3Mn1−xAlx (x = 0–0.1) ceramics showed no presence of phase impurities and/or essential compositional inhomogeneities at the boundaries. The grain size varies in the range 1–5 μm; representative examples of SEM micrographs of frac Sr0.7Ce0.3Mn0.9Al0.1O3−δ are given in Fig. 2. The dilatometric studies of Sr0.7Ce0.3Mn1−xAlx (x = 0–0.2) materials demonstrated that their expansivity 100–830 °C in air is almost linear (Fig. 3). For x slight increase in the thermal expansion coefficient is observed at temperatures above 610–630 °C. This behaviour may be associated with the transition of composition from tetragonally distorted to cubic perovskite.
### Table 1

Properties of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_{x}\text{O}_{3-\delta}$ ceramics

<table>
<thead>
<tr>
<th>Composition</th>
<th>Unit cell parameters</th>
<th>Activation energy for total conductivity</th>
<th>Thermal expansion coefficients</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$a$ (nm)</td>
<td>$c$ (nm)</td>
<td>$T$ (°C)</td>
</tr>
<tr>
<td>$\text{Sr}<em>{0.7}\text{Ce}</em>{0.3}\text{MnO}_3$</td>
<td>0.3838(4)</td>
<td>0.3860(7)</td>
<td>350–850</td>
</tr>
<tr>
<td>$\text{Sr}<em>{0.7}\text{Ce}</em>{0.3}\text{Mn}<em>{0.9}\text{Al}</em>{0.1}\text{O}_3$</td>
<td>0.3830(8)</td>
<td>0.3854(9)</td>
<td>360–850</td>
</tr>
<tr>
<td>$\text{Sr}<em>{0.7}\text{Ce}</em>{0.3}\text{Mn}<em>{0.8}\text{Al}</em>{0.2}\text{O}_3$</td>
<td>0.3820(6)</td>
<td>0.3832(8)</td>
<td>340–850</td>
</tr>
</tbody>
</table>

The average TEC values are listed in Table 1. The substitution of manganese decreases TEC values down to $10.8 \times 10^{-6}$ K$^{-1}$, which provides a good compatibility with commonly used solid electrolytes based on zirconia and ceria. Note that the TEC values of alternative mixed-conducting cathodes, including $(\text{La, Sr})(\text{Fe, Co})\text{O}_3$, are significantly higher than that of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{1-x}\text{Al}_x\text{O}_{3-\delta}$ and close to incompatibility with stabilized zirconia solid electrolytes.

#### 3.2. Oxygen permeation

Selected results of the oxygen permeation measurements are summarized in Figs. 4 and 5. As expected, the steady-state permeation fluxes ($j$) through $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_3$ membranes decrease with increasing membrane thickness ($d$) (Fig. 4A). However, such a decrease is considerably lower than predicted by the integral form of Wagner equation. This is accompanied with increasing specific oxygen permeability.
be expected due to well-known correlations between the exchange currents and bulk oxygen diffusion.\textsuperscript{28}

The relative roles of the bulk ambipolar transport and surface exchange may be estimated decomposing the overall driving force, expressed as $\ln(p_2/p_1)$, into three contributions associated with membrane bulk and surfaces. The contributions to overcome limitations of surface exchange kinetics can be written as $\ln(p_2/p_2')$ for the feed side, with an oxygen partial pressure drop from $p_2$ to $p_2'$, and $\ln(p_1'/p_1)$ for the permeate side, with a drop from $p_1'$ to $p_1$. The driving force to overcome the resistance to bulk ion transfer is, respectively, $\ln(p_2'/p_1')$. One may express the flux as $j = k_{ex} \ln(p_2/p_2') = k_{ex}' \ln(p_1'/p_1)$, where $k_{ex}$ is the exchange coefficient, and the superscripts “f” and “p” denote the feed and permeate sides, correspondingly. Under steady-state conditions, the permeation flux through the membrane bulk, $j = (RT/16F^2d)\sigma_{amb} \ln(p_2'/p_1')$, is equal to the fluxes through the membrane/gas boundaries. This yields

$$\frac{\ln(p_2/p_1)}{j} = \left[\frac{16F^2}{RT\sigma_{amb}}\right] \left(\ln(p_2/p_2') + \frac{k_{ex}'}{k_{ex}}\right)$$

where $p_1$ and $p_2$ are the oxygen partial pressures at the membrane permeate and feed sides, respectively. Such a behaviour unambiguously indicates that the overall oxygen transport is limited both by the bulk ambipolar conductivity and oxygen surface exchange kinetics; the role of the latter factor decreases when the membrane thickness increases.\textsuperscript{20,21,28} A significant surface effect is also characteristic for undoped Sr\textsubscript{0.7}Ce\textsubscript{0.3}Mn\textsubscript{0.8}Al\textsubscript{0.2}O\textsubscript{1.9} membranes.\textsuperscript{14}

Fig. 6 presents the temperature dependencies of the permeation fluxes through Sr\textsubscript{0.7}Ce\textsubscript{0.3}Mn\textsubscript{0.8}Al\textsubscript{0.2}O\textsubscript{1.9} under fixed oxygen chemical potential gradient. Increasing aluminum concentration increases oxygen transport, whereas the apparent activation energies are essentially independent of Al content. This suggests an increase in the oxygen nonstoichiometry, caused by the incorporation of acceptor-type A\textsuperscript{3+}.

As a result, the ionic ($\sigma_{ion}$) and ambipolar ($\sigma_{amb}$) conductivities increase on doping, whilst the energetic barrier for ion migration seems essentially unchanged. On the other hand, an enhancement in the surface exchange kinetics might also

\[ \text{J(O}_2\text{)} = \frac{kd}{(\ln(p_2/p_1))^{-1}} \]
suit, the total conductivity decreases almost linearly with aluminum additions (inset in Fig. 9). The incorporation of 20% Al\(^{3+}\) into the manganese sublattice lowers the conductivity by a factor of about 0.5. Nevertheless, the level of \(\sigma\) values for \(\text{Sr}_0.7\text{Ce}_0.3\text{Mn}_{1-x}\text{Al}_x\text{O}_3-\delta\) is still in the order of 120–250 S cm\(^{-1}\) at 700–950 °C, which is sufficient to provide low Ohmic losses at the cathode.\(^{31}\)

In the low-temperature range, the values of activation energy (\(E_a\)) for the total conductivity are relatively low and nearly independent of Al content, within the limits of experimental uncertainty (Table 1). The activation energy decreases with increasing temperature, and an apparent transition from semiconducting to pseudo-metallic behaviour is observed at temperatures above approximately 800 °C. The negligible dependence of the activation energy on aluminum concentration suggests that Al\(^{3+}\) cations do not alter significantly the electronic transport mechanism.

3.3. Total conductivity in air

The total conductivity (\(\sigma\)) in the system \(\text{Sr}_0.7\text{Ce}_0.3\text{Mn}_{1-x}\text{Al}_x\text{O}_3-\delta\) decreases with increasing dopant content (Fig. 9). The difference between total and ionic conductivity values (Figs. 8 and 9) unambiguously shows that the electronic contribution is predominant; the ion transference number values are lower than 10\(^{-4}\). The substitution of Mn cations with Al\(^{3+}\) having a constant oxidation state, leads to partial blocking of electronic transport, which occurs via the Mn\(^{4+}\)–O–Mn\(^{3+}\) bonds.\(^{29,30}\) As a result, the total conductivity decreases almost linearly with aluminum additions (inset in Fig. 9). The incorporation of 20% Al\(^{3+}\) into the manganese sublattice lowers the conductivity by a factor of about 0.5. Nevertheless, the level of \(\sigma\) values for \(\text{Sr}_0.7\text{Ce}_0.3\text{Mn}_{1-x}\text{Al}_x\text{O}_3-\delta\) is still in the order of 120–250 S cm\(^{-1}\) at 700–950 °C, which is sufficient to provide low Ohmic losses at the cathode.\(^{31}\)

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3.4. Total conductivity and Seebeck coefficient versus \(p(O_2)\)

The negative sign of the Seebeck coefficient of (Sr, Ce)MnO\(_{3-\delta}\) has been interpreted as an indication that electronic transport is n-type.\(^{12}\) However, the pseudo-metallic conductivity behaviour at high temperatures (Fig. 9) suggests a decrease in charge carrier concentration, as found mostly for perovskite compounds with p-type behaviour.\(^{8,17,21,23,29,30}\)

Moreover, when a single mechanism of the electronic transport is prevailing, the Seebeck coefficient (\(\alpha\)) can be described by a generic approximate solution using the ratio (\(C_R\)) between charge carrier concentration and the density of states: \(^{32,33}\)

\[
\alpha \approx \pm \frac{k}{e} \ln \left( \frac{1}{C_R} - 1 \right) \quad (3)
\]

with positive sign for p-type (\(C_R = p/N\)) and negative for n-type (\(C_R = n/N\)) behaviour, where \(N\) is the density of states. Obviously, one cannot distinguish the cases when \(C_R = n/N < 0.5\) or \(C_R = p/N > 0.5\). For manganites with predominant p-type conduction, one usually ascribes the concentration of electron holes to Mn\(^{4+}\) content,\(^{29,30}\) whereas Hashimoto and Iwahara\(^{12}\) considered trivalent manganese cations as n-type charge carriers in (Sr, Ce)MnO\(_{3-\delta}\), neglecting the contributions of Ce\(^{3+/4+}\) and Mn\(^{2+}\) formed due to disproportionation. In these two cases, the corresponding \(C_R\) values are \(p/N \approx [\text{Mn}^{4+}]/[\text{Mn}]\) or \(n/N \approx 1 - [\text{Mn}^{4+}]/[\text{Mn}]\), where \([\text{Mn}]\) is the total manganese concentration. Both hypotheses yield

\[
[Mn^{4+}] = \frac{[\text{Mn}]}{1 + \exp(\alpha e/k)} \quad (4)
\]

Eq. (4) shows that considering the thermopower sign is insufficient to distinguish n-type or p-type behaviour; an analysis of the charge carrier concentration as a function of temperature and oxygen pressure is necessary for these goals. In particular, whilst the n-type electronic conductiv-
Fig. 10. Oxygen partial pressure dependencies of the total conductivity of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ under oxidizing conditions.

Fig. 11. Oxygen partial pressure dependencies of the Seebeck coefficient of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ under oxidizing conditions.

Fig. 12. Temperature dependencies of the $p/N$ ratio and estimated mobility for the p-type and n-type electronic charge carriers in $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ at $p(\text{O}_2) = 1.58 \times 10^{-3}$ atm.

Fig. 13. Oxygen pressure dependencies of the $p/N$ ratio and estimated mobility for the p-type and n-type electronic charge carriers in $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ at 1173 K.

Fig. 14. Oxygen partial pressure dependencies of the Seebeck coefficient of $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ under oxidizing conditions.

Fig. 15. Temperature dependencies of the $p/N$ ratio and estimated mobility for the p-type and n-type electronic charge carriers in $\text{Sr}_{0.7}\text{Ce}_{0.3}\text{Mn}_{0.9}\text{Al}_{0.1}\text{O}_{1.8}$ at 1173 K.
the μ values tend to increase slightly with increasing oxygen chemical potential, probably due to increasing average oxidation state of Mn cations, with a corresponding decrease in their size and crystal lattice contraction. As a result, greater overlapping of manganese and oxygen electron orbitals causes a stronger covalence of the Mn—O—Mn bonds and higher degree of the electron delocalization. Similar trends are well known for other perovskite oxides.

3.5. Phase stability

The perovskite phase stability limits under reducing conditions can be estimated from the abnormal drop in the total conductivity at low p(O₂) (Fig. 14). For example, at 900 °C, such a drop starts at p(O₂) ≈ 2.6 × 10⁻¹⁴ atm and is maximum at approximately 10⁻¹⁵ atm. After reduction, strong hysteresis effects on redox cycling are observed, as illustrated by the inset in Fig. 14. These variations of the conductivity indicate decomposition of the perovskite-type phase, confirmed by XRD. One example of the XRD pattern of Sr₀.₇Ce₀.₃Mn₀.₉Al₀.₁O₃₋δ quenched after annealing in reducing atmosphere at 900 °C, is presented in Fig. 1. When temperature decreases, the decomposition occurs at lower oxygen chemical potentials, as expected.

Fig. 15 shows the approximate phase stability boundary of Sr₀.₇Ce₀.₃Mn₀.₉Al₀.₁O₃₋δ, evaluated from the conductivity and Seebeck coefficient data. The stability limits of Mn₃O₄, LaMnO₃ and two iso-concentration lines of CeO₂₋y are given for comparison. The decomposition of Sr₀.₇Ce₀.₃Mn₀.₉Al₀.₁O₃₋δ perovskite occurs at oxygen pressures close to the iso-nonstoichiometry line of CeO₂₋y with y = 0.009, suggesting that the change in the oxidation state of cerium cations within the stability domain of Sr₀.₇Ce₀.₃Mn₀.₉Al₀.₁O₃₋δ should be minor. Moreover, the estimations of Mn⁴⁺ fraction from the Seebeck coefficient using Eq. (4), in combination with the crystal electroneutrality and site conservation conditions, suggest that no less than 60% cerium cations are trivalent even under oxidizing conditions. Also, the ionic radii of Ce⁴⁺ and Mn⁴⁺ yield better geometrical matching for A- and B-site cations in the perovskite structure, i.e. a tolerance factor closer to unity, when compared to smaller Ce⁴⁺ cations in the A sublattice and larger Mn⁴⁺ in the B sites. The decomposition of doped strontium manganite is therefore associated with changes of the manganese oxidation state on reduction, as for LaMnO₃₋δ. Most likely, the formation of substantial amounts of Mn³⁺ leads to excessive stress in the lattice, followed by structural collapse.

4. Conclusions

Aluminum-substituted Sr₀.₇Ce₀.₃MnO₃₋δ exhibits a comparatively high oxygen ionic conduction, phase stability in a relatively wide range of conditions, sufficient level of the electronic transport, and thermal expansion compatible with that of common solid electrolytes. These properties are attractive for possible application of Sr₀.₇Ce₀.₃Mn¹₋ₓAlₓO₃₋δ for SOFC cathodes. Partial substitution of Mn by Al enhances the oxygen permeability and ionic conductivity. However, as the solubility of Al³⁺ in the lattice of strontium manganite is quite low, additional doping with aliovalent cations may be desirable in order to achieve higher oxygen ionic conductivity, closer to that of solid-electrolyte materials. The dependencies of total conductivity and Seebeck coefficient on oxygen partial pressure and temperature are indicative of prevailing hole transport, with a slight conductivity increase on increasing p(O₂) and a transition from semiconducting to pseudo-metallic behaviour at temperatures above 800 °C.
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