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Conductivity and stability of ceramic $\text{Sr}_{1-x}\text{Y}_x\text{FeO}_{3-\delta}$ solid solutions

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Abstract

The total electric conductivity of two ceramics of cubic perovskites corresponding to $x = 0.1$ and $x= 0.2$ in the $\text{Sr}_{1-x}\text{Y}_x\text{FeO}_{3-\delta}$ solid solution has been measured by a 4-probe method in the temperature range from 298 K to 1373 K in air, allowing activation energies of $\approx 0.1 - 0.2$ eV to be extracted. The effect of the oxygen partial pressure ($P_{O2}=0.21 – 10^{-15}$ atm) on the conductivity of the $\text{Sr}_{0.8}\text{Y}_{0.2}\text{FeO}_{3-\delta}$ ceramic has also been investigated at high temperatures, i.e. at 1123 K, 1223 K and 1323 K. By analyzing the X-ray diffraction patterns of the quenched powder samples, the $P_{O2}$-stability of $\text{Y}_{0.2}\text{Sr}_{0.8}\text{FeO}_{3-\delta}$ has been determined. These results are discussed in terms of the defect structure in $\text{Sr}_{1-x}\text{Y}_x\text{FeO}_{3-\delta}$, which accounts well for the present data and especially the sign change of the majority charge carriers as $P_{O2}$ is reduced. This study presents important new features of the total conductivity with respect to temperature and oxygen non-stoichiometry in these ceramics.

Keywords: Y-doped strontium ferrite; Conductivity of ceramic; Thermodynamic stability; Defect structure

1. Introduction

Recently, mixed Sr and rare earth (RE) ferrites with the $\text{Sr}_{1-x}\text{RE}_x\text{FeO}_{3-\delta}$ formula that possess the perovskite type structure have been investigated as cathodes for solid oxide fuel cells.
(SOFCs), membranes, catalysts, and sensors [1-7]. Special attention has been paid to the Sr-rich yttrium-containing ferrites [8-10]. Indeed, the oxidation of Fe$^{3+}$ in air-prepared REFeO$_3$ oxygen stoichiometric orthoferrites to Fe$^{4+}$ in SrFeO$_3$ requires high-oxygen-pressure techniques, implying the stability of Fe$^{3+}$ [11]. This is confirmed by the SrFe$^{3+}$O$_{2.5}$ brownmillerite preparation conditions (first firing at 1673K in air followed by a second one at 1573K in vacuum [12]). In this respect, the strontium rich Sr$_{1-x}$RE$_x$FeO$_{3-δ}$ (x>0.5) oxides prepared in air are of special interest due to a large concentration of oxygen vacancies, as well as a significant fraction of Fe$^{4+}$ ions within a disordered cubic structure. In contrast to the brownmillerite phase, the latter as a rule exhibits enhanced mobility of oxygen and higher conductivity values. This was shown more recently in a detailed study of phase equilibria in the $\frac{1}{2}$Y$_2$O$_3$ – SrO – $\frac{1}{2}$Fe$_2$O$_3$ system where the introduction of even a small amount of yttrium into strontium ferrite was found to stabilize the cubic perovskite structure of Sr$_{1-x}$Y$_x$FeO$_{3-δ}$ solid solutions (0.05≤x≤0.25) [13].

However, information concerning the functional properties of these Sr$_{1-x}$Y$_x$FeO$_{3-δ}$ ceramic complex oxides and their stability at low oxygen partial pressure is still very limited. Indeed, the only conductivity measurements previously reported were in air and N$_2$ atmospheres for 0<x≤0.37 [8], as a function of P$_{O2}$ for x=0.33 [10], in air and Ar atmospheres for x=0.33 [14], and in air for x=0.00, 0.25, 0.50, 0.75 [15]. Therefore, a study of the solid solution Sr$_{1-x}$Y$_x$FeO$_{3-δ}$ (x = 0.1, 0.2) has been undertaken. In the following, we report on conductivity measurements as a function of temperature and oxygen partial pressure, as well as the thermodynamic stability of these ceramic samples at low oxygen pressure.

2 Experimental

The samples of Sr$_{1-x}$Y$_x$FeO$_{3-δ}$ (x = 0.1, 0.2) were prepared using the glycerol-nitrate technique. Yttrium oxide Y$_2$O$_3$, strontium carbonate SrCO$_3$ and iron oxalate FeC$_2$O$_4$×2H$_2$O were used as starting materials. Y$_2$O$_3$ and SrCO$_3$ were annealed before weighing in order to remove adsorbed gases and water. The starting reagents, taken in the appropriate ratios, were dissolved
in nitric acid. Then glycerol in the amount needed for the complete reduction of nitrate ions was added. Heating of the solution first led to the formation of a viscous gel that subsequently transformed to powder. The powder was slowly heated within the temperature interval of 298 – 1273 K. The final annealing was performed at 1373 K for 100 – 120 h with intermediate regrinding in alcohol after each 10 – 12 h. For the conductivity measurements, dense ceramic samples of \( \text{Sr}_{1-x} \text{Y}_x \text{FeO}_{3-\delta} \) were prepared in the form of 3×3×25 mm bars by powder pressing followed by sintering at 1573 K in air for 24 h, with subsequent slow cooling at the rate of 1 K/min. The densities of the polished ceramic samples were 90 – 95 % of their theoretical values calculated from the XRD data.

Phase purity was examined by powder x-ray diffraction (XRD) using a Shimadzu XRD 7000 diffractometer in Cu-K\( \alpha \) radiation (\( \lambda = 1.5418 \, \text{Å} \)) with a pyrolytic graphite monochromator within the angular range 20°≤2\( \Theta \)≤90° (scan step 0.02° or 0.04° with exposure time 2–10 s).

Conductivity measurements were performed by a standard 4-probe method using mechanically pressed contacts of platinum wire. Temperature (300 \( \leq T \) (K) \( \leq 1400 \)) and oxygen partial pressure (0.21 \( \leq P_{O_2} \) (atm) \( \leq 10^{-16} \)) inside a cell equipped with a YSZ oxygen pump and oxygen sensor were controlled using a Zirconia-318 device [16].

3. Results and discussion

XRD results confirmed the formation of single-phase \( \text{Sr}_{1-x} \text{Y}_x \text{FeO}_{3-\delta} \) (x = 0.1, 0.2) with cubic structure (SG \( Pm\overline{3}m \)) in accordance with the results reported earlier [13]. The XRD pattern for single-phase \( \text{Sr}_{0.8} \text{Y}_{0.2} \text{FeO}_{3-\delta} \) refined by the Rietveld analysis is shown in Fig. 1, as an example. The refined unit cell parameters are: \( a=3.875(1) \, \text{Å} \) for \( \text{Sr}_{0.9} \text{Y}_{0.1} \text{FeO}_{3-\delta} \) and \( a=3.865(1) \, \text{Å} \) for \( \text{Sr}_{0.8} \text{Y}_{0.2} \text{FeO}_{3-\delta} \).

3.1. Conductivity of ceramic \( \text{Sr}_{1-x} \text{Y}_x \text{FeO}_{3-\delta} \) (x = 0.1, 0.2) in air

The temperature dependence of the conductivity of \( \text{Sr}_{1-x} \text{Y}_x \text{FeO}_{3-\delta} \) (x = 0.1, 0.2) in air (Fig. 2) show a shape typical for the rare earth ferrites and cobaltites [8, 17-21]. The conductivity value \( \sigma = 9.3 \, \text{S} \cdot \text{cm}^{-1} \) at 373 K for \( \text{Sr}_{0.8} \text{Y}_{0.2} \text{FeO}_{3-\delta} \) (the lowest temperature in our study) practically
coincides with that reported in [22] (for the highest in their temperature range). Since the oxygen content in \( \text{Sr}_{1-x}\text{Y}_x\text{FeO}_{3-\delta} \) \((x = 0.1, 0.2)\) remains constant while heating from the room temperature up to 600 K [13], it can be inferred that the concentration of charge carriers has not changed either. Thus, the increase of the low-temperature conductivity (up to 600 K) for both compounds appears due to the increase in charge-carrier mobility, since it is thermally activated (see inset in Fig. 2). The subsequent decrease in conductivity at higher temperature was explained by oxygen release from oxide [16, 17] and the concomitant drop of the charge-carrier concentration. The maximum in the value of the conductivity at 600 K (Fig. 2) correlates well with the beginning of oxygen release from the crystal lattice of \( \text{Sr}_{1-x}\text{Y}_x\text{FeO}_{3-\delta} \) \((x = 0.1, 0.2)\) [13]. The increasing concentration of oxygen vacancies causes a decrease in the concentration of holes, which serve as the majority of mobile charge carriers [17-22]. The oxygen vacancies can also decrease the mobility of holes since their presence breaks some of the (Fe – O – Fe) pathways along which charge carriers migrate [17]. The mean oxidation state of iron at room temperature (measured by the iodometric titration method described in [13]) is 3.38 for a sample with \( x=0.1 \), which is larger than 3.10 for \( x=0.2 \) [13]. This explains the higher concentration of holes in the former and therefore the higher conductivity of \( \text{Sr}_{0.9}\text{Y}_{0.1}\text{FeO}_{3-\delta} \) as compared to \( \text{Sr}_{0.8}\text{Y}_{0.2}\text{FeO}_{3-\delta} \) oxide. In the low-temperature region (below the conductivity maximum) where the concentration of charge carriers is constant, the conductivity is thermally activated (see the inset in Fig. 2). Data in this temperature range was used to calculate activation energies by considering the formula for conductivity within the small polaron model expressed as : 

\[
\sigma = \frac{A}{T} \exp \left( - \frac{E}{RT} \right).
\]

The extracted values of activation energy are in the range 0.1 to 0.2 eV for both compositions, which is typical for the hopping conductivity mechanism.

3.2. \( \text{PO}_2 \) stability of \( \text{Sr}_{0.8}\text{Y}_{0.2}\text{FeO}_{3-\delta} \)

Prior to the study of the conductivity of \( \text{Sr}_{0.8}\text{Y}_{0.2}\text{FeO}_{3-\delta} \) versus oxygen partial pressure, we examined its thermodynamic stability while decreasing \( \text{PO}_2 \). A sample of \( \text{Sr}_{0.8}\text{Y}_{0.2}\text{FeO}_{3-\delta} \) was annealed at fixed \( T \) and \( \text{PO}_2 \) in the environmentally isolated cell, which allows the sample to be
quenched to room temperature without being opened. Fig. 3 illustrates the XRD data for Sr_{0.8}Y_{0.2}FeO_{3-δ} processed at various T and P_{O_2}. In contrast to undoped SrFeO_{3-δ}, which is transformed to the brownmillerite structure at some point (for example, at 10^{-3} atm, 700ºC [23]), a single phase Sr_{0.8}Y_{0.2}FeO_{3-δ} cubic structure is observed to remain under all the conditions studied. The unit cell parameters of the Sr_{0.8}Y_{0.2}FeO_{3-δ} single phase after various heat treatments are listed in Table 1.

It can be seen from Table 1 that the unit cell parameter a increases with a decrease in the oxygen partial pressure and with an increase in temperature. Such behavior can be explained by increasing oxygen deficiency, which leads to a decrease in the oxidation state of iron, and hence an increase in the radius of the Fe ions. It is worth noting that the largest unit cell parameter belongs to the sample that was quenched from 1373 K in air. This shows that the temperature influence on both oxygen nonstoichiometry and unit cell expansion is stronger than a decrease in oxygen partial pressure. However more precise analysis of this effect requires a detailed examination of oxygen nonstoichiometry versus oxygen partial pressure.

### 3.3. Conductivity of ceramic Sr_{0.8}Y_{0.2}FeO_{3-δ} versus P_{O_2}

Fig. 4 illustrates a (P_{O_2})-conductivity dependence for Sr_{0.8}Y_{0.2}FeO_{3-δ}. Since a detailed study of oxygen nonstoichiometry versus oxygen partial pressure is unavailable, we perform a simplified qualitative analysis. In order to analyze the shape of conductivity curves versus oxygen partial pressure in terms of the defect structure [24], a reference “stoichiometric” oxide has to be chosen. Since the concentration of Fe^{3+} ions in Sr_{0.8}Y_{0.2}FeO_{3-δ}, which should be treated as a ground state, is predominant within the entire range of temperature and P_{O_2}, the most convenient composition that can be taken as an “ideal” stoichiometric point is YFe^{3+}O_3, the only difference being the lack of Sr in the A sites of the perovskite structure.

Assuming that the oxygen content remains equal to 3, the partial substitution of Sr for Y will induce changes of the iron oxidation state according to the following
In terms of the quasi-chemical approach, Sr for Y substitution in YFe$_3^+$O$_3$ can be written as follows:

$$2SrO + Fe_2O_3 + \frac{1}{2}O_2 \rightarrow 2Sr'_Y + 2Fe'_{Fe} + 6O'_O$$  \hspace{1cm} (1),

where here and below we use the Kröger and Vink notation, in which Fe$^{4+}$, Fe$^{3+}$ and Fe$^{2+}$ are denoted as Fe', Fe$^\times$ and Fe' respectively. The equilibrium constant for this reaction is described by the following equation:

$$K_1 = [Sr'_Y]^2[Fe'_{Fe}]^2[O'_O]^{-\frac{1}{2}}P_{O_2}^{-\frac{1}{2}}$$  \hspace{1cm} (2).

At the same time significant oxygen losses and hence the formation of oxygen vacancies ($V'_O$) causes a decrease in the concentration of Fe$^{4+}$ ions (Fe') according to the following quasi-chemical reaction:

$$O'_O + 2Fe'_{Fe} \leftrightarrow \frac{1}{2}O_2 + V'_O + 2Fe'_{Fe}$$  \hspace{1cm} (3).

The equilibrium constant for this reaction can be written as follows:

$$K_3 = \frac{[V'_O][Fe'_{Fe}]}{[O'_O][Fe'_{Fe}]}\frac{1}{2}P_{O_2}$$  \hspace{1cm} (4).

As a result the formula of oxide should be written as: Sr$_{0.8}$Y$_{0.2}$Fe$^{4+}$Fe$^{3+}$$_{0.8-2\delta}$O$_{3+8\delta}$.  

It is widely acknowledged that a complete description of the defect structure of the rare earth ferrites and their derivatives, together with atomic disordering in the oxygen sublattice must also include the charge disproportionation process [17, 25]:

$$2Fe'_{Fe} \leftrightarrow Fe'^{\times}_{Fe} + Fe'_{Fe}$$  \hspace{1cm} (5).

For which the relevant equilibrium constant is:

$$K_4 = \frac{[Fe'^{\times}_{Fe}][Fe'_{Fe}]}{[Fe'_{Fe}]}$$  \hspace{1cm} (6).

The combination of equations (3) and (5) yields one more equation:

$$O'_O + 2Fe'_{Fe} \leftrightarrow \frac{1}{2}O_2 + V'_O + 2Fe'_{Fe}$$  \hspace{1cm} (7).

$$K_7 = \frac{[V'_O][Fe'_{Fe}]}{[O'_O][Fe'_{Fe}]}\frac{1}{2}P_{O_2}$$  \hspace{1cm} (8).
Indeed, it was observed that the mean oxidation state of iron at high temperature in air reaches 3+ and even lower values [13]. Thus, at low oxygen partial pressure, Fe$^{3+}$ ($\text{Fe}^\times$) and Fe$^{2+}$ ($\text{Fe}'$) are expected to be predominant iron states.

Taking into account all defect species, the total electroneutrality condition can be written as follows:

$$[\text{Sr}_\gamma'] + [\text{Fe}^\prime_{\text{Fe}e}] = [\text{Fe}'_{\text{Fe}e}] + 2[V^\circ_0]$$  \hspace{1cm} (9)

Bearing in mind that electronic-type conductivity is predominant in this type of oxides, Fe$^{4+}$ and Fe$^{2+}$ can be treated as holes and electrons localized on Fe$^{3+}$ ions respectively, i.e. $[\text{Fe}^\prime_{\text{Fe}e}] = p$ and $[\text{Fe}^\prime_{\text{Fe}e}] = n$.

In the vicinity of the minimal conductivity value it can be considered that $n \approx p$ [24], and hence from the electroneutrality condition one can deduce following equality $[\text{Sr}_\gamma'] \approx 2[V_0^\circ]$. With this approximation, it is easy to show from equations (4) and (8) that $n \sim P_{\text{O}_2}^{-\frac{1}{4}}$ and $p \sim P_{\text{O}_2}^{\frac{1}{4}}$.

The total electrical conductivity is proportional to the concentration of charge carriers

$$\sigma = (z_i e) \times B_i \times [i]$$ \hspace{1cm} (10)

where $B_i$ is the mobility of charge carriers, which remains unchanged at constant temperature, and therefore $\sigma \sim P_{\text{O}_2}^{-\frac{1}{4}}$ at $P_{\text{O}_2} < (P_{\text{O}_2})_{\text{min}}$, and $\sigma \sim P_{\text{O}_2}^{\frac{1}{4}}$ at $P_{\text{O}_2} > (P_{\text{O}_2})_{\text{min}}$. Despite all these assumptions, this $P_{\text{O}_2}$ dependence of $\sigma$ is found to fit to the experimental data (Fig. 4).

Further increase of $P_{\text{O}_2}$ leads to the assumption that $p >> n$. Within this approximation the electroneutrality condition can be approximated by the form:

$$[\text{Sr}_\gamma'] = 0.8 = [\text{Fe}^\prime_{\text{Fe}e}] + 2[V_0^\circ]$$ \hspace{1cm} (11)

It is worth noting that even in air, the concentration of oxygen vacancies in Sr$_{0.8}$Y$_{0.2}$FeO$_{3-\delta}$ is still significant, since the oxygen content ($3-\delta$) at room temperature is equal to 2.68 [11]. Eq. (11) means that the increase in the concentration of holes leads to a decrease in the concentration of oxygen vacancies, and therefore to a first approximation $[V_0^\circ]$ and $[\text{Fe}^\prime_{\text{Fe}e}]$ are inversely proportional.
The reaction of oxygen incorporation into the lattice can be written as follows:

\[
\frac{1}{2}O_2 + V^{\circ}_O + 2Fe^{\circ}_{Fe} \leftrightarrow O^{\circ}_O + 2Fe^{\circ}_{Fe}
\]  

and the equilibrium constant for reaction (13) is:

\[
K_4 = \frac{[O^{\circ}_O][Fe^{\circ}_{Fe}]^2}{[V^{\circ}_O][Fe^{\circ}_{Fe}]^2} P^{-1}\frac{1}{O_2}
\]

Assuming that changes of oxygen nonstoichiometry at fixed temperature are not so significant compared to the oxygen content, i.e. \((3-\delta) \approx Const,\) as well as \([Fe^{\circ}_{Fe}] \approx Const,\) and taking into account expression (12) it is easy to deduce that the concentration of holes \(p\) in the oxide is proportional to \(P^{\delta}\). Thus the \(\sigma-(P_{O_2})\) dependence according to equation (10), in the region where the predominant charge carriers are holes, has to follow the relationship \(\sigma \sim P^{\delta}\). Indeed the slope of the \(\lg \sigma = f(\lg P_{O_2})\) dependence decreases to a value of 1/6 as the oxygen partial pressure approaches air condition (Fig. 4). This effect is more visible at lower temperature (1123K in Fig. 4) since the equilibrium constant (6) for the disproportionation process (5) rises with increasing temperature.

4 Conclusions

The present study of two ceramics, \(Sr_{0.9}Y_{0.1}FeO_{3-\delta}\) and \(Sr_{0.8}Y_{0.2}FeO_{3-\delta}\), belonging to the \(Sr_{1-x}Y_xFeO_{3-\delta}\) solid solution, allowed several important conclusions to be drawn. The compounds of this series crystallizing in the cubic perovskite structure, and not the brownmillerite structure, remain stable even at a relatively low oxygen partial pressure (down to \(10^{-16}\) atm) and high temperature (1123 – 1323 K). The total conductivity \((\sigma)\) of \(Sr_{1-x}Y_xFeO_{3-\delta}\) in air is found to decrease with increasing \(Y\) content. This is explained by a decrease in \(Fe^{4+}\) content and hence of the \(p\)-type charge carrier concentration. As \(T\) increases, \(\sigma\) goes through a maximum near 600K, a temperature below which the conductivity can be fitted by a small polaron model, leading to an
activation energy that decreases as $x$ decreases. As revealed by the $\lg \sigma = \lg (P_{O2})$ plots collected at $T$ as high as 1323K, the oxygen partial pressure decrease induces a sign change of the predominant charge carriers in $\text{Sr}_{0.8}Y_{0.2}\text{FeO}_{3-\delta}$ from $p$ in air to $n$ at low $P_{O2}$. These experimental results, as well as the slope of the conductivity curves in the regions of low and high $P_{O2}$, corresponding to $n$ and $p$-type carriers, respectively, are in good agreement with the data and defect model reported by Poulsen et al. [21] and Kozhevnikov et al. [23] for undoped strontium ferrite.

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**Figure Captions**

**Fig. 1** Rietveld refinement (*SG Pm3m*) results of XRD data at room temperature for Sr$_{0.8}$Y$_{0.2}$FeO$_{3-\delta}$. Experimental data: symbols; calculated profile: continuous line; allowed Bragg reflections: vertical marks. The difference between the experimental and calculated profiles is displayed at the bottom of the graph.

**Fig. 2.** Total electrical conductivity of Sr$_{1-x}$Y$_x$FeO$_{3-\delta}$ (x=0.1; 0.2) ceramics versus temperature in air. Inset: $\ln(\sigma \times T)$ as a function of $1/T$ (lower x-scale) and $T$ (upper x-scale) for the low $T$ part of the $\sigma(T)$ curves; the lines are for Arrhenius from which activation energy values are obtained.

**Fig. 3** Room temperature XRD patterns for Sr$_{0.8}$Y$_{0.2}$FeO$_{3-\delta}$ treated at different conditions labelled in the graph. The indexation and positions (vertical marks) of diffraction peaks are also given (*SG Pm3m*).

**Fig. 4** Total electrical conductivity of Y$_{0.2}$Sr$_{0.8}$FeO$_{3-\delta}$ ceramics versus oxygen partial pressure for 3 T labelled in the figure. The exponents $n$ are for the calculated slopes of the $\sigma \sim P^{n}_{O_2}$ expressions derived from the defect structure analysis (see text) ; they are indicated in the graph with the corresponding calculated lines.

**Table 1** – The unit cell parameters for Sr$_{0.8}$Y$_{0.2}$FeO$_{3-\delta}$ after various heat treatment

<table>
<thead>
<tr>
<th>$a$, Å</th>
<th>Heat treatment details</th>
<th>lg(Po$_2$), atm</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.865(1)</td>
<td>Slowly cooled to room temperature</td>
<td>– 0.68, air</td>
</tr>
<tr>
<td>3.950(1)</td>
<td>Quenched from 1373 K</td>
<td>– 0.68, air</td>
</tr>
<tr>
<td>3.882(1)</td>
<td>Quenched from 1123 K</td>
<td>– 13</td>
</tr>
<tr>
<td>3.879(1)</td>
<td>Quenched from 1223 K</td>
<td>– 11</td>
</tr>
</tbody>
</table>
$Y_{0.2}Sr_{0.8}FeO_{3.8}$

slowly cooled from 1373 K in air

$R_p=8.3\%$, $R_B=9.7\%$, $R WP=13\%$, $R exp=8.78\%$, $R_I=7.76\%$
$Y_{0.2}Sr_{0.8}FeO_{3-\delta}$
quenched from 1223 K, $P_{O_2}$/atm=10

$R_p=8.6\%$, $R_{Br}=8.3\%$, $R_{wp}=11\%$, $R_{exp}=6.71\%$, $R_t=8.76\%$
$Y_{0.2}Sr_{0.8}FeO_{3.8}$
quenched from 1123 K, $P_{O_2}/atm=10^{-13}$
$R_p=9.9\%, R_B=9.2\%, R_{wp}=13\%,$
$R_{exp}=7.93\%, R_I=8.1\%$
Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐ The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

On behalf of authors Vladimir Cherepanov