Temperature dependence and gas-sensing response of conduction for mixed conducting SrFeyCozOx thin films
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Mixed conducting ceramic materials of the type SrFe<sub>y</sub>Co<sub>2</sub>O<sub>x</sub> are characterized by their oxygen nonstoichiometry and their high electronic and ionic conductivities. Consequently, they are suitable candidate materials for applications such as oxygen separation membranes, electrode materials for solid oxide fuel cells, and solid-state chemical sensors. It has been reported that some SrFe<sub>y</sub>Co<sub>1</sub>O<sub>x</sub> materials have oxygen ion mobilities greater than yttria-stabilized zirconia (YSZ) below 800°C. Recently, the high oxygen ion mobility of the SrFe<sub>0.5</sub>Co<sub>1.5</sub>O<sub>x</sub> (1.0 < γ < 1.5) compositions has been attributed to the perovskite-type SrFe<sub>Co</sub>O<sub>1</sub> phase.

It has previously been shown that perovskite-based ceramic materials which exhibit p-type semiconductor and mixed conductivity may be suitable candidates for temperature-independent gas sensors. Williams et al. examined a series of ferrates including BaFe<sub>1</sub>−<sub>y</sub>TaO<sub>3</sub>, which showed negligible conductivity dependence to temperature, between 600 and 800°C. More recently, Moos et al. demonstrated that acceptor- and donor-doped SrTi<sub>1</sub>−<sub>y</sub>FeO<sub>1</sub>−<sub>x</sub> materials could be tailored to yield temperature-independent conductivity responses between 700 and 900°C. These materials were being investigated for applications such as oxygen sensors for combustion exhaust control.

Thick-film perovskite materials were used for all these studies, and the temperature-independent regions were above 500°C. Thin films provide advantages related to the higher surface area-to-volume ratio. Thinner films of gas sensors based on a bulk equilibrium mechanism between the film and the gas mixture provide faster response times. Previous work has shown that it is possible to grow high-quality thin films of SrFeO<sub>x</sub> (2.5 < x < 3.0) using pulsed laser deposition (PLD), which shows oxygen-sensing properties between 350 < T < 500°C.

For some gas-sensing applications, it would be advantageous to use transducer materials which exhibit a temperature-independent resistance response at lower temperatures and still demonstrate adequate gas sensitivity. In this study, SrFe<sub>Co</sub>O<sub>x</sub> materials grown as thin films are shown to have reversible bulk oxygen equilibration properties which may be exploited to achieve a conductivity response for O<sub>2</sub>, CO, and H<sub>2</sub> sensing, which has a negligible temperature dependence.

**Experimental**

All SrFe<sub>y</sub>Co<sub>2</sub>O<sub>x</sub> materials were prepared as sintered pellets using standard ceramic techniques. High-purity (~99.99%) SrCO<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Co<sub>3</sub>O<sub>4</sub> materials were mixed in the required ratios, followed by thermal treatments in flowing argon and/or oxygen at 1050-1100°C. These materials were then pressed into 12 mm diameter targets followed by sintering at 1000-1150°C under flowing oxygen. The pellets were all verified to be single phase by X-ray diffraction (XRD) before being used as targets for pulsed laser ablation deposition (PLD). The X-ray spectra were indexed according to previously determined crystal structures. SrFe<sub>Co</sub>O<sub>x</sub> (γ = 0.25, 0.5, 0.75, 0.9, 1.0) were indexed to the cubic perovskite structure, SrCoO<sub>x</sub> to the hexagonal structure, and SrFe<sub>X</sub>Co<sub>2</sub>O<sub>x</sub> and SrFe<sub>Co</sub>O<sub>x</sub> to the rhombohedral structure.
SrFe$_2$Co$_{y}$O$_{x}$ films were both indexed to an orthorhombic phase, which is an intergrowth of repeating layers of perovskite and rock-salt layers.9,12,30

Two series of SrFe$_2$Co$_{y}$O$_{x}$ thin films on (1102) single-crystal sapphire substrates were prepared by laser deposition using a Lambda-Physik LPX305i laser operating at $\lambda = 248$ nm. These included the solid solution perovskite series SrFe$_{2-x}$Co$_{x}$O$_{3}$ (y = 0, 0.25, 0.5, 0.75, 0.9, 1.0) and the structurally related materials SrFe$_{1.3}$Co$_{0.7}$O$_{3}$ and SrFe$_{1.5}$Co$_{0.5}$O$_{3}$. For each composition, the films were deposited at two separate thicknesses, approximately 30 and 300 nm. A laser pulse rate of 8 Hz was used with an energy density at the target of 1.5 J cm$^{-2}$. During the deposition step, the sapphire substrate was heated at 700°C under a background gas of 13 Pa oxygen, followed by cooling at approximately 10°C/min in a background oxygen pressure of 53 kPa.

X-ray spectra for both pellets and films were collected using a Cu Kα Scintag XDS2000 diffractometer with a secondary beam graphite monochromator over the range 20 = 2° to 80°. The very intense peaks from the single-crystal sapphire substrate also showed satellite peaks arising from W-L$_{α}$ and W-L$_{β}$ X-rays originating from the W filament in the X-ray tube. The very weak peak at 20 = 43.2° (Fig. 1) is attributable to such a satellite peak. Film thicknesses were estimated based on stylus profiler measurements taken with a Dektak IIA instrument on masked 300 nm SrFe$_{2}$Co$_{y}$O$_{x}$ films grown on sapphire. Elemental analysis for targets and films were performed by inductively coupled plasma atomic emission spectroscopy (ICP-AES) using standard solutions to determine the ratio of Sr, Fe, and Co elements. Scanning electron microscopy (SEM) images were taken of the as-deposited films using a JEOL JSM 5300 instrument.

Electrical conductivity measurements of the films were taken using a two-wire method in a controlled environment where the temperature could be varied between 20 and 600°C and the composition of the flowing gas changed as required. High-purity ($\{O_2\} + [N_2] > 99.99\%$) gas mixtures flowing at 220 cm$^3$/min in a 1 L volume chamber were used for all tests. Electrical contacts were achieved by thermally depositing 200 nm Au pads directly onto the SrFe$_{2}$Co$_{y}$O$_{x}$ films. Sheet resistances and conductivities of the films were calculated from the resistance measurement and corrected for the geometrical configuration of the Au electrode pads and the thickness of the film.31 Experimental details are provided elsewhere.23,25

In order to determine the dynamic temperature dependences, the resistances of the films were measured while cooling at a controlled rate of 10°C/min from 500 to 100°C. Prior to this, the films were equilibrated at 500°C for at least 3 h in one of two flowing O$_2$/N$_2$ gas mixtures: 0.2% O$_2$ or 100% O$_2$. The composition of the flowing gas mixture was invariant throughout the cooling cycle. For each film, this procedure was repeated at least twice to ensure reproducibility. The conductivities of the films as a function of the O$_2$/N$_2$ gas mixtures were measured isothermally at 500°C. This was done by measuring the film resistances as the O$_2$/N$_2$ gas mixtures were varied from oxygen-poor (~0.01% O$_2$) to oxygen-rich (100% O$_2$) conditions in a stepwise manner, typically waiting 60 min before changing to the next O$_2$/N$_2$ gas composition. The gas mixtures were then changed in the reverse order from oxygen-rich to oxygen-poor in order to verify reversibility.

**Results and Discussion**

**Film composition and structure.**—XRD analysis of the 300 nm films deposited on sapphire showed the films to be crystalline and textured (Fig. 1). No reflections for the 30 nm films were observed except for the very strong sapphire reflections from the substrate due to the small sample thickness of these films. The SrFe$_{2}$Co$_{1-y}$O$_{x}$ (y = 0.5, 0.75, 0.9, 1.0) films were all indexed to the cubic perovskite structure26 and were (110) textured. The SrFe$_{0.75}$Co$_{0.25}$O$_{3}$ film showed an additional degree of (111) texturing. The SrCo$_{2}$O$_{4}$ film (Fig. 1f) was indexed to the SrCo$_{2}$O$_{4}$ hexagonal structure and was textured (201).25 It was not clear from the SrFe$_{1.5}$Co$_{0.5}$O$_{3}$ film (Fig. 1e) whether it adopted the cubic perovskite or the hexagonal structure. Likewise, the SrFe$_{1.3}$Co$_{0.7}$O$_{3}$ and SrFe$_{1.5}$Co$_{0.5}$O$_{3}$ films could not be unambiguously assigned to the expected orthorhombic phase, since the reflection at 20 = 32.7° could be indexed to either the (110) reflection of the cubic perovskite structure or to the composite (400) + (002) or (251) reflections of the orthorhombic phase.23,25,30

SEM analysis of all the 30 nm films showed the films to be featureless and smooth at a length scale $\approx 0.1$ μm. The 300 nm films showed grain boundaries and/or scales at a length scale (l) of 1 $\leq l \leq 5$ μm for SrFe$_{1-y}$Co$_{y}$O$_{3}$ (y = 0.25, 0.5, 0.75, 0.9, 1.0) compositions. For example, micrographs of the 300 nm SrFe$_{0.75}$Co$_{0.25}$O$_{3}$ film clearly showed 1–3 μm cracks or scales (Fig. 2). The SrCo$_{2}$O$_{4}$, SrFe$_{1.3}$Co$_{0.7}$O$_{3}$, and SrFe$_{1.5}$Co$_{0.5}$O$_{3}$ films showed no evidence of scaling but showed some roughness at a length scale...
of 0.1 ≤ ℓ ≤ 0.3 μm. Elemental analysis of the films showed that the Sr, Fe, and Co metallic elements occurred in the correct ratios to within the 5% estimated error limits of the ICP-AES technique. XRD, elemental, and SEM analyses confirmed that PLD is a suitable technique for the stoichiometric transfer of material from the target to the substrate for heterometallic oxides such as mixed conducting SrFe₃Co₃O₁₀ ceramics.

**Dependence of temperature on film conductivity.**—The conductance response to temperature for two 30 nm thick films (SrFeOₓ and SrFe₃Co₃O₁₀) is shown in Fig. 3 as a set of Arrhenius plots. The higher conductivity of both films in 100% O₂ compared to 0.2% O₂ confirms that these materials are p-type semiconducting oxides. It is also evident that the conductance response to temperature is much smaller for SrFe₃Co₃O₁₀ than for SrFeOₓ, especially in the temperature range 200 ≤ T ≤ 500°C. An activation energy parameter, E_A, related to this temperature dependence of film conductance has been extracted from the Arrhenius plots of the entire SrFe₃CoO₁₋ₓOₓ series of films. The results summarized in Table I show the E_A values which were calculated for two temperature ranges. The 350 ≤ T ≤ 500°C “nonstoichiometric” range was chosen since at these temperatures, bulk oxygen equilibration of the film is possible. Consequently, the balancing of the thermal activation of charge carriers effect with the “nonstoichiometric” effect can occur (Eq. 1-2). By comparison, the 100 ≤ T ≤ 200°C “stoichiometric” range was chosen since the oxygen stoichiometry for the films is expected to be quenched when the experimental cooling rate of 10°C/min was used.

In general, a higher level of cobalt substitution results in smaller temperature dependencies, with the smallest E_A values reported for the SrFe₀.₇₅Co₀.₂₅Oₓ, SrFe₀.₅Co₀.₅Oₓ, and SrFe₀.₃Co₀.₇₅Oₓ films. It is also noteworthy that the temperature dependences of the films exposed to 0.2% O₂ are generally greater than those exposed to 100% O₂. This effect of decreased temperature dependence with oxygen content is consistent with other p-type perovskite materials including SrFeOₓ2532 and (Ba,Sr)Fe₁₋ₓTaₓO₃.18-21

A significant influence of film thickness on the temperature dependence is also evident from Table I. In the nonstoichiometric region, 350 ≤ T ≤ 500°C, reduced temperature dependence for the thinner films is indicated by smaller E_A values. Some of these values are even negative, indicating that a decreased temperature actually results in an increased conductance. This phenomenon has previously been reported for other mixed oxide perovskites, albeit at higher temperatures18-21. Figure 4 illustrates how the film thickness can influence the temperature-dependent conduction response. The thinner 30 nm film has a smaller temperature dependence over a larger temperature range. This may be the result of a higher surface area-to-volume ratio for the thinner film, resulting in faster bulk oxygen equilibration, or to an effect related to the degree of interfacial stress between the substrate and the film.

**Table I.** Activation energies of conduction, E_A, for SrFe₃Co₃O₁₀ films on sapphire in the “nonstoichiometric” (350-500°C) and “stoichiometric” (200-100°C) temperature ranges. E_A values were derived from dynamic 10°C/min temperature cooling ramps.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Thickness (nm)</th>
<th>100% O₂</th>
<th>0.2% O₂</th>
<th>100% O₂</th>
<th>0.2% O₂</th>
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<tr>
<td>SrFeOₓ</td>
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<td>0.37</td>
<td>0.19</td>
<td>0.28</td>
<td>0.49</td>
</tr>
<tr>
<td>SrFe₃Co₀.₁₅Oₓ</td>
<td>30</td>
<td>0.10</td>
<td>0.12</td>
<td>0.22</td>
<td>0.39</td>
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<tr>
<td>SrFe₀.₇₅Co₀.₂₅Oₓ</td>
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<td>0.20</td>
<td>-0.04</td>
<td>0.18</td>
<td>0.31</td>
</tr>
<tr>
<td>SrFe₀.₅Co₀.₅Oₓ</td>
<td>30</td>
<td>0.13</td>
<td>0.10</td>
<td>0.12</td>
<td>0.26</td>
</tr>
<tr>
<td>SrFe₀.₃Co₀.₇₅Oₓ</td>
<td>30</td>
<td>0.04</td>
<td>-0.003</td>
<td>0.12</td>
<td>0.26</td>
</tr>
<tr>
<td>SrCoOₓ</td>
<td>30</td>
<td>0.08</td>
<td>0.12</td>
<td>0.08</td>
<td>0.31</td>
</tr>
<tr>
<td>SrCo₃O₇</td>
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<td>0.06</td>
<td>0.02</td>
<td>0.15</td>
<td>0.18</td>
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<tr>
<td>SrCo₅O₉</td>
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<td>-0.005</td>
<td>0.17</td>
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<td>0.21</td>
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<td>SrCo₉O₁₂</td>
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<td>0.31</td>
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<tr>
<td>SrCo₁₀O₁₃</td>
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<td>0.02</td>
<td>0.10</td>
<td>0.19</td>
<td>0.32</td>
</tr>
<tr>
<td>SrCo₁₁O₁₄</td>
<td>30</td>
<td>0.07</td>
<td>0.064</td>
<td>0.19</td>
<td>0.29</td>
</tr>
</tbody>
</table>

**Figure 3.** Arrhenius plots for two 30 nm films in different O₂/N₂ mixtures: SrFe₀.₇₅Co₀.₂₅Oₓ in (a) 100% and (c) 0.2% O₂; and SrFeOₓ in (b) 100% and (d) 0.2% O₂.

**Figure 4.** Effects of film thickness on the conductance response with temperature for two SrFe₃Co₃O₁₀ films: (●) 30 and (▽) 300 nm. Cooling ramps were carried out in 100% O₂ at 10°C/min.
ing, chemical equilibrium between the film and the surrounding gas mixture cannot be maintained as the kinetics of O₂ exchange slows down and quenching occurs. The temperature at which quenching occurs is dependent on both film composition, thickness, and cooling rate. Figure 5 shows the resistance response when a SrFe₀.₂₅Co₀.₇₅O₃ film is first equilibrated in air at three different temperatures between 200 and 350°C in air, cooled by 25°C at 10°C/min, followed by re-equilibration at the new temperature. At higher temperatures (Fig. 5a) the system maintains equilibrium throughout the 10°C/min cooling rate. Cooling results in an immediate decrease in film resistance resulting from the dominant role of the nonstoichiometric effect (Eq. 2). When the temperature is at an intermediate range (Fig. 5b), the resistance of the film initially increases with decreasing temperature, as would be expected on the basis of thermally activated charge carriers. Once the final temperature is reached, the resistance no longer continues to increase but instead decreases. This is due to the nonstoichiometric effect, where the film continues to react with the oxygen from the surrounding gas mixture, resulting in an increased number of hole charge carriers. At this intermediate temperature range, bulk equilibration still occurs, although too slow for equilibrium to be maintained during the cooling step. At lower temperatures (Fig. 5c), quenching occurs and the oxygen stoichiometry of the film does not change. As a result, the balancing of thermal effects cannot occur, and the material behaves as a normal semiconductor, where decreasing temperature results in increased resistance.

Oxygen-sensing properties of SrFeₓCo₁₋ₓO₃ thin films.—The effects on conductivity of varying the O₂/N₂ composition at T = 500°C was examined for all of the SrFeₓCo₁₋ₓO₃ films. Figure 6 shows a typical example where the composition of the O₂/N₂ gas mixture was changed stepwise from 0.2% O₂ to 100% O₂ and back to 0.2% O₂. At 500°C, the resistance response to variations in oxygen composition is rapid and reversible. The response is faster when the O₂ concentration is increasing compared to when the O₂ concentration is decreasing. The response time is determined in part by the relatively slow mixing time for the 220 cm³/min gas mixtures in the 1 L volume of the chamber. The results of the isothermal variation of O₂/N₂ gas mixtures for the SrFeₓCo₁₋ₓO₃ set of films are summarized in Fig. 7-10. The results for the SrFe₀.₇Co₀.₃O₃ and SrFe₁₋ₓCoₓO₃ films are summarized in Fig. 11.

In all cases, exposure to increasing oxygen concentration levels resulted in increased film conductivity, as would be expected for p-type semiconducting oxides. Film conductivities at 500°C ranged between 20 and 200 S/cm for films exposed to 100% O₂ atmosphere and 0.4-25 S/cm for films exposed to 0.01% O₂. The conductivity of the films increased with increasing cobalt substitution of the SrFeO₃ parent material up to a maximum value. This maximum occurred within the range of composition SrFeₓCo₁₋ₓO₃ (0.25 ≤ x ≤ 0.75) and was dependent on film thickness and the composition of the gas mixture. The SrFe₁₋ₓCoₓO₃ and SrFe₁₋ₓCoₓO₃ set of films also showed that cobalt substitution resulted in increased conductivity. There were some subtle differences between the 30 nm series (Fig. 7 and 9) and the 300 nm (Fig. 8 and 10) series of films, including differences in the composition where maximum conductivities were observed. This is possibly an effect of the increased role of interfacial stress between the film and the substrate, or is due to the increasing contribution of the space-charge regions at the interfaces for the thinner films.

The sensitivity of the film to oxygen with respect to its change in conductivity may be represented as the slope of the log(conductivity) vs. log(pO₂) plot. This slope corresponds to a parameter 1/N, where N is a constant determined by the dominant type of bulk defect involved in the equilibrium between oxygen and the
sensitivity is observed for both the SrFe films is shown in Fig. 12. For the 300 nm films, high oxygen sen-

sion, and SrFeO has also previously been shown that SrFeO films may used to detect reduc-

Enhanced oxygen sensitivity of SrFeO has been previously attributed to phase transitions between the brownmillerite and cubic perovskite phases. At 500°C this phase transition is expected to occur at 0.005 atm. Liu et al. have also identified a similar phase transition for SrFeO below 750°C for 0.05 atm. The sensitivity of the films to oxygen between 0.002 atm. The speed of response is relatively slow at 300°C. The time required for 90% of the change of resistance to occur (t90)

0.5

Figure 9. The effects of cobalt substitution and O2/N2 gas composition (% O2) on conductivity for a series of 30 nm SrFeO films at 500°C.

Figure 10. The effects of cobalt substitution and O2/N2 gas composition (% O2) on conductivity for a series of 300 nm thick SrFeO films at 500°C.

SrFeO, whereas it is only observed for SrFeO for the 30 nm films. This demonstrates that film thickness influences to some extent, the sensitivity of the film to oxygen.

Sensing reducing gases with SrFeO thin films.—It has previously been shown that SrFeO films may used to detect reducing gases in air by monitoring the change in film resistance with gas exposure. This is also the case for SrFeO thin films. Figure 13 shows the resistance response of a 30 nm SrFeO film to exposure to H2, CO, and CH4 in background air at 300°C. The film shows good sensitivity to H2, moderate sensitivity to CO, and negligible sensitivity to CH4. The operating temperature of 300°C was selected for this film because in a background atmosphere of air, this film exhibits very little temperature dependence for its resistivity. The speed of response is relatively slow at 300°C.

Figure 11. Conductivity of SrFeO films at 500°C for different O2/N2 gas compositions and different thicknesses. 1/N represents different slopes of the log(σ) vs. log ρO2 relationship: (a) SrFeO 30 nm; (b) SrFeO 300 nm; (c) SrFeO 30 nm; and (d) SrFeO 300 nm.

Figure 12. Conductivity of SrFeO films at 500°C for different O2/N2 gas compositions and different thicknesses. 1/N represents different slopes of the log(σ) vs. log ρO2 relationship: (a) SrFeO 30 nm; (b) SrFeO 300 nm; (c) SrFeO 30 nm; and (d) SrFeO 300 nm.
Resistance response at 300°C of a 30 nm SrFeO$_{1-x}$O$_x$ series not normally associated with gas sensors based on bulk equilibration of the film is typically between 10 and 15 min. The slow response time is a consequence of its dependence on the bulk equilibration of the film and not normally associated with gas sensors based on bulk equilibration, which normally operates above 500°C. Moreover, at this temperature, the film is operating in nearly a temperature-independent region.

Conclusions

It has been demonstrated that thin films of SrFeO$_{1-x}$O$_x$ grown by PLD exhibit novel conductivity responses with respect to variation in both temperature and gas composition. Due to the balancing of thermal effects, it is possible to obtain a conduction response which has a negligible temperature dependence between 200 $\leq T \leq$ 500°C. This effect was most pronounced for the perovskites with compositions SrFe$_{0.5}$Co$_{0.5}$O$_3$ and SrFe$_{0.25}$Co$_{0.75}$O$_3$. Thinner films (30 nm) show a greater temperature independence than the thicker (300 nm) films at temperature between 350 $\leq T \leq$ 500°C. All the SrFeCoO$_x$ films showed reversible p-type gas sensor responses at 500°C to variations in O$_2$/N$_2$ gas mixtures. Regions of enhanced oxygen sensitivity over narrow $p_{O_2}$ ranges were attributed to phase transitions between the cubic perovskite and brownmillerite phases. Finally, it was shown that it is possible to use SrFeCoO$_x$ films to detect H$_2$ and CO in background air while operating under conditions where there is negligible temperature dependence.

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References


Figure 12. Effects of cobalt substitution on oxygen sensitivity at 500°C for the SrFeCo$_{1-x}$O$_x$ series of films at two thicknesses: (a) 30 and (b) 300 nm. Sensitivities were calculated between 0.2 and 2% O$_2$.

Figure 13. Resistance response at 300°C of a 30 nm SrFe$_{0.5}$Co$_{0.5}$O$_3$O$_x$ film to (a) 1% CO; (b) 1000 ppm CO$_2$; (c) 1% H$_2$; and (d) 1000 ppm CH$_4$ in background air.