## ORIGINAL PAPER

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# Stabilities and electrical conductivities of electrode materials for use in H<sub>2</sub>S-containing gases

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Abstract Stabilities and conductivities of sulfides and lithiated sulfides derived from La<sub>0.9</sub>Sr<sub>0.1</sub>Ga<sub>0.8</sub>Cr<sub>0.2</sub>O<sub>3</sub>,  $LaCr_{0.9}Ti_{0.1}O_3$ ,  $Y_{0.9}Ca_{0.1}FeO_3$ , and  $SrCo_{0.8}Fe_{0.2}O_3$ are studied in a gas mixture of 96% H2 and 4% H2S at 900 °C using X-ray diffraction, microscopy, and impedance spectroscopy. Results indicate that the sulfide and lithiated sulfide derived from Y<sub>0.9</sub>Ca<sub>0.1</sub>FeO<sub>3</sub> exhibit not only high stability but also high electrical conductivity, implying that they are viable candidate electrode materials for H<sub>2</sub>S polishing and for solid oxide fuel cells using H<sub>2</sub>S-containing fuels. In contrast, the lithiated sulfides derived from La<sub>0.9</sub>Sr<sub>0.1</sub>Ga<sub>0.8</sub>Cr<sub>0.2</sub>O<sub>3</sub> and LaCr<sub>0.9</sub> Ti<sub>0.1</sub>O<sub>3</sub> exhibit relatively low electrical conductivity, although their chemical and thermal stability appear to be very good. On the other hand, the lithiated sulfides derived from SrCo<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3</sub> show inadequate chemical stability under the testing conditions even though the electrical conductivity appears to be good.

**Key words** Electrochemical hydrogen sulfide polishing · Electrode stability · Conductivity · Sulfide · Lithiated sulfide

#### Introduction

Fuel cells have emerged as a leading technology to provide advanced power sources in an environmentally clean and efficient manner. All current fuel cell systems, however, have very limited tolerance to hydrogen sul-

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J. Winnick School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332-0100, USA fide; the H<sub>2</sub>S concentration in the feed derived from fossil resources must be less than 1 ppm for optimum operation. To reach this goal, an efficient and economical H<sub>2</sub>S polishing process is needed. An electrochemical cell similar in design to a molten carbonate fuel cell (MCFC) has been developed to remove H<sub>2</sub>S from hot reformate streams at high temperatures and convert it directly to H<sub>2</sub> and a separate stream of elemental sulfur vapor (e.g. [1, 2, 3]). The cathodic and anodic reactions involved in the one-step, continuous process can be described, respectively, as:

$$H_2S + 2e^- \to H_{2(g)} + S^{2-}$$
 (1)

and

$$S^{2-} \to \frac{1}{2}S_2 + 2e^-$$
 (2)

and the overall reaction is

$$H_2S \to \frac{1}{2}S_2 + H_2$$
 (3)

This process is not only efficient but also modular and compact owing to its simplicity of operation compared with conventional H<sub>2</sub>S removal through adsorption or chemical reactions [1, 2, 3]. While electrochemical H<sub>2</sub>S polishing has been studied for more than 10 years, many critical issues still remain. For example, an efficient and stable cathode material needs to be identified. Although preliminary results showed that Ni, Fe, and Co sulfides exhibit acceptable electrochemical performance, their long-term stabilities are yet to be demonstrated [2]. Ni and Co sulfides have low melting points, especially the lithiated sulfides, which limits the operating temperature of the H<sub>2</sub>S removal system. The melting points of iron sulfides (>1144 °C) are higher than those of Ni and Co sulfides; the use of iron sulfides derived from iron oxide as electrodes for this process, however, led to short-circuiting of the cell, which was due probably to the high solubility of iron ions in the carbonate electrolytes within the membrane [2]. The solubilities of Ni and Co ions are also considerable in the carbonate electrolytes [4]. Further, the electrodes for H<sub>2</sub>S polishing are usually operated under severe reducing environments; simple composition metal sulfides have a tendency to be reduced at temperatures well below their melting points, which may lead to a significant change in electrode microstructure.

Because of the problems encountered in using simple sulfides or lithiated sulfides as electrodes for H<sub>2</sub>S polishing, we here describe better electrode materials with high stability and conductivity. As reported recently [5, 6, 7, 8, 9, 10, 11], complex or composite oxides usually exhibit high conductivity and stability over a wide range of oxygen partial pressures; they have been studied widely as electrolyte or electrodes for solid oxide fuel cells (SOFCs) and as mixed-conducting membranes for gas separation. The materials exhibiting high conductivity and stability under reducing conditions may be used as electrodes for H<sub>2</sub>S polishing. They may also offer needed H<sub>2</sub>S tolerance as anodes in SOFCs.

LaGaO<sub>3</sub>-based materials are promising electrolytes for medium-temperature solid state ionic devices because of their high ionic conductivity and adequate stability under reducing conditions [5, 6]. The substitution of Ga by Cr in  $\text{La}_{1-x}\text{Sr}_x\text{GaO}_3$  is an effective way to further increase the electronic conductivity.

LaCrO<sub>3</sub>-based materials also have high stability over a wide range of oxygen partial pressures. To date, Cadoped LaCrO<sub>3</sub> is still the most promising bipolar interconnect material for SOFCs [7, 9]. Mg-doped LaCrO<sub>3</sub> has also been investigated as a cathode material for H<sub>2</sub>S polishing by Weaver and Winnick [2]. Their primary results showed that LaCrO<sub>3</sub> has high stability but low conductivity and little catalytic activity. The addition of a donor impurity such as Ti into LaCrO<sub>3</sub> can increase the electrical conductivity greatly under strong reducing conditions, as reported by Karen and Norby [7]. Thus, doped LaCrO<sub>3</sub> could be a good alternative as an electrode for H<sub>2</sub>S polishing.

Y<sub>0.9</sub>Ca<sub>0.1</sub>FeO<sub>3</sub> has been proposed by Kim and Yoo [10] as a cathode material for SOFCs. It exhibits high ionic as well as electronic conductivity within the oxygen partial pressure range of 10<sup>-16</sup> atm to 1 atm. It was reported that the charge carriers in this material depended strongly on the partial pressure of oxygen. The behavior of this material in hydrogen sulfide is yet to be characterized. SrCo<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3</sub> is also a promising mixed conductor, having been used as a membrane reactor for methane oxidation.

The electrodes for H<sub>2</sub>S polishing may react with lithium carbonate electrolyte under operating conditions, leading to changes in chemical composition and

electrochemical properties. The final composition of the electrode is determined by both the kinetics as well as thermodynamics of the reactions among sulfides, lithium carbonate, and gas reactant. In this paper, the stability and conductivity of  $La_{0.9}Sr_{0.1}Ga_{0.8}Cr_{0.2}O_3$ ,  $LaCr_{0.9}Ti_{0.1}O_3$ ,  $Ca_{0.1}Y_{0.9}FeO_3$ , and  $SrCo_{0.8}Fe_{0.2}O_3$  exposed to  $H_2S$  with or without lithium carbonate and potassium carbonate are investigated.

#### **Experimental**

Sample preparation

The electrode materials  $La_{0.9}Sr_{0.1}Ga_{0.8}Cr_{0.2}O_3$ ,  $LaCr_{0.9}Ti_{0.1}O_3$ ,  $Y_{0.9}Ca_{0.1}FeO_3$ , and  $SrCo_{0.8}Fe_{0.2}O_3$  were prepared using a traditional ceramic process. The precursors were  $La_2O_3$  (99.99%, from Aldrich),  $TiO_2$  (99.9%, from Aldrich),  $Sr(NO_3)_2$  (99%, from Alfa),  $Ga_2O_3$  (99.999%, from Alfa),  $Y_2(CO_3)_3$ .3H $_2O$  (99.9%, from Alfa),  $Fe(NO_3)_3$ .9H $_2O$  (98–101%, from Alfa),  $Co(CH_3CO_2)_2$ .4H $_2O$  (99.9% from Alfa),  $CrO_3$  (99–99.5%, from Fisher), and  $CaCO_3$  (99.5%, from Fisher). Stoichiometric amounts of precursors were mixed and ball milled in ethanol (with zirconia balls in a plastic bottle) for 24 h, followed by drying and calcination at 1100 °C in air for 12 h. The phase compositions of the calcined powders were examined using X-ray diffraction (XRD, Philips PW 1800 with Cu  $K\alpha$  radiation). The resulting products with desired phase compositions were again ball milled in ethanol for 24 h to reduce the particle size.

The prepared oxides were then exposed to a gas mixture of 96% H<sub>2</sub> and 4% H<sub>2</sub>S at 900 °C for different periods of time (6, 40 h). The resulting products are called "sulfides", although some of the oxides may not be completely converted to sulfides. XRD was used to identify the phase compositions of the resulting products.

To prepare lithiated sulfides, the prepared oxides were mixed with  $(Li_{0.62}K_{0.38})_2CO_3$ ; the mixture was then treated in a gas mixture of 96%  $H_2$  and 4%  $H_2S$  at 900 °C for different periods of time (6, 40 h). The weight ratio of the oxide to the carbonate was kept at 2:1. After treatment, the mixtures were washed several times with boiling water to remove the carbonates remaining in the mixture. The resulting products are called "lithiated sulfides". All the sulfides and lithiated sulfides prepared were fine powders with melting points higher than 900 °C under the gas mixture of 96%  $H_2$  and 4%  $H_2S$ . The names of the oxides, sulfides, and lithiated sulfides are abbreviated as shown in Table 1.

#### Conductivity measurements

The conductivities of the samples calcined at 900 °C for 6 h were measured using impedance spectroscopy. All impedance spectra were acquired using an EG & G 273 potentiostat/galvanostat and a 5201 lock-in amplifier interfaced with a personal computer. The frequency was scanned from 0.01 Hz to 100 kHz; the amplitude of the applied alternating perturbation was 5 mV. Unless stated otherwise, the samples used for conductivity measurements were rectangular bars cut from pellets pressed at 4000 psi and sintered at 900 °C in 96% H<sub>2</sub> and 4% H<sub>2</sub>S for 2 h. The dimension of the bars was about  $15 \times 2 \times 2$  mm; 1 wt% polyvinyl alcohol (PVA) was used as binder for the pressing of pellets. Pt paste was painted on

Table 1 The abbreviations used for the oxides, sulfides, and lithiated sulfides

Туре	La <sub>0.9</sub> Sr <sub>0.1</sub> Ga <sub>0.8</sub> Cr <sub>0.2</sub> O <sub>3</sub>	LaCr <sub>0.9</sub> Ti <sub>0.1</sub> O <sub>3</sub>	$Y_{0.9}Ca_{0.1}FeO_3$	SrCo <sub>0.8</sub> Fe <sub>0.2</sub> O <sub>3</sub>
Oxide	LSGC-O	LCT-O	CYF-O	SFC-O
Sulfide	LSGC-S	LCT-S	CYF-S	SFC-S
Lithiated sulfide	Li-LSGC-S	Li-LCT-S	Li-CYF-S	Li-SFC-S

the surfaces of both ends of each bar and a platinum grid was attached and pressed on each Pt electrode with a spring loading system. Pt lead wires were connected to the Pt grids. The bar samples were placed in a quartz tube and exposed to a gas mixture of 96% H<sub>2</sub> and 4% H<sub>2</sub>S during impedance measurements.

The bulk resistance of each cell  $(R_{\rm cell})$  was obtained from the intercept of the impedance arc on the x-axis at high frequencies, whereas the resistance of the Pt wires and grids  $(R_{\rm l})$  was determined from an impedance measurement of a blank cell with the two Pt grids shorted. The bulk resistance of each sample,  $R_{\rm b}$ , is then given by:

$$R_{\rm b} = R_{\rm cell} - R_1 \tag{4}$$

and the conductivity of each sample,  $\sigma$ , was calculated as follows:

$$\sigma = \frac{1}{R_{\rm b}} \left( \frac{L}{S} \right) \tag{5}$$

where L is the distance between the two electrodes on the bar and S is the area of the electrode (the cross section of the sample).

The electrical measurements were conducted at 650, 690, 730, 770, and 810 °C in dry and wet gas mixtures of 96%  $\rm H_2$  and 4%  $\rm H_2S$ . The wet gas mixtures were produced by flowing through a water bubbler kept at 25 °C. The flow rate of the inlet gas was about 100 mL min<sup>-1</sup>.

The microstructures and morphologies of the pellets were further characterized using a scanning electron microscope (SEM, Hitachi S-800).

#### **Results and discussion**

 $La_{0.9}Sr_{0.1}Ga_{0.8}Cr_{0.2}O_3$ 

As shown in Fig. 1, the XRD spectrum of LSGC-O resembles the pattern of LaGaO<sub>3</sub>; some small peaks corresponding to SrO can also be observed. The XRD spectrum of LSGC-O remains unchanged when exposed to 96% H<sub>2</sub> and 4% H<sub>2</sub>S at 900 °C for 6 h. When the treatment time was increased to 40 h, several small peaks corresponding to SrLaGa<sub>3</sub>OS<sub>6</sub> were observed. Peaks of LSGC-O remain the same during the treatment with

Fig. 1 XRD patterns of LSGC-O, LSGC-S, and Li-LSGC-S. The time in hours adjacent to each spectrum represents the time that the sample was exposed to the gas mixture of 96% H<sub>2</sub> and 4% H<sub>2</sub>S at 900 °C

96%  $H_2$  and 4%  $H_2S$ . The addition of  $(Li_{0.62}K_{0.38})_2CO_3$  to LSGC-S leads to the appearance of the phase of La<sub>2</sub>O<sub>2</sub>S after sintering in 96%  $H_2$  and 4%  $H_2S$  at 900 °C for 6 h. There is no further phase change after sintering the samples up to 40 h.

Shown in Fig. 2 are the conductivities of LSGC-S and Li-LSGC-S in dry and wet 96% H<sub>2</sub> and 4% H<sub>2</sub>S. It can be seen that the conductivities of Li-LSGC-S are higher than those of LSGC-S in both dry and wet gas mixtures. The conductivities of the two materials decreased after the addition of water into the inlet gas. The activation energy of the two samples was about 40 kJ mol<sup>-1</sup> in the dry gas, and it increased slightly after the addition of

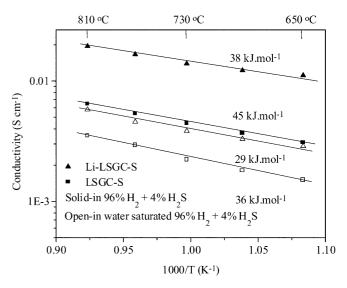
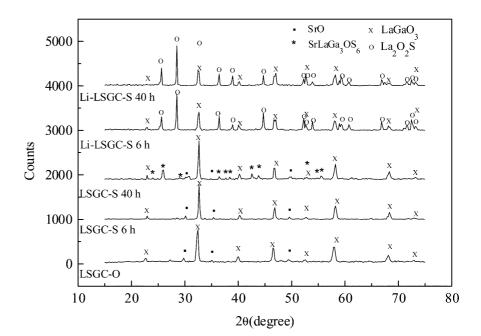
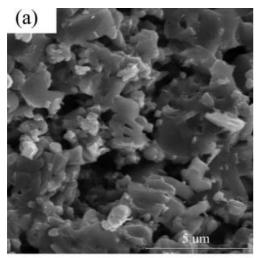


Fig. 2 Arrhenius plots of conductivities as measured in dry or humidified gas mixtures (96%  $H_2+4\%$   $H_2S$ ) of LSGC-S and Li-LSGC-S pellets sintered at 900 °C for 2 h. The number adjacent to each line represents the activation energy for electrical conductivity



water vapor. However, the observed conductivities of LSGC-based sulfides and lithiated sulfides (e.g.,  $0.01~\rm S~cm^{-1}$  at 650 °C) seem to be inadequate as electrodes for  $\rm H_2S$  polishing. The characteristics of the electrical conduction in LSGC-O exposed to air were investigated by Baker et al. [5] and it was concluded that the electronic conduction is p-type. However, the conductivity data shown in Fig. 2 seem to suggest that the electronic conduction in LSGC-S and Li-LSGC-S exposed to  $\rm H_2S$  and  $\rm H_2$  is n-type, because the conductivities measured in wet gases (with higher  $P_{\rm O_2}$ ) were much smaller than those measured in dry gases (with lower  $P_{\rm O_2}$ ). Further studies are needed to reveal the details of the conduction mechanism.

Shown in Fig. 3 are some typical SEM micrographs of LSGC-S and Li-LSGC-S pellets fired at 900 °C in 96% H<sub>2</sub> and 4% H<sub>2</sub>S for 2 h. It appears that LSGC-S melts slightly at 900 °C, as can be seen in Fig. 3a. However, Li-LSGC-S is porous with a porosity about 40%, as determined from Fig. 3b. The SEM micrographs and the XRD patterns suggest that Li-LSGC-S



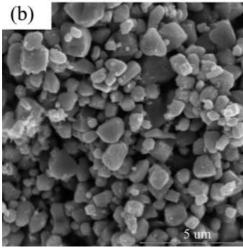


Fig. 3 SEM cross-sectional views of as-sintered pellets of (a) LSGC-S and (b) Li-LSGC-S sintered at 900 °C for 2 h in dry 96%  $\rm H_2+4\%~H_2S$ 

has good thermal and chemical stability up to 900 °C under the conditions for H<sub>2</sub>S polishing.

Because of its low conductivity, Li-LSGC-S alone may not be suitable as an electrode for H<sub>2</sub>S polishing despite its good chemical and thermal stability. However, Li-LSGC-S could be a viable component of a composite electrode for H<sub>2</sub>S polishing. For example, Li-LSGC-S may be used as a supporting substrate for low-melting-point electrodes with high conductivity and catalytic activity. It is a natural extension of the concept of supported catalyst in heterogeneous catalysis.

 $LaCr_{0.9}Ti_{0.1}O_3$ 

Shown in Fig. 4 are the XRD patterns of LCT-O, LCT-S, and Li-LCT-S. It seems that the LCT-O is quite stable in 96% H<sub>2</sub>+4% H<sub>2</sub>S up to 900 °C for 40 h with or without lithium carbonate. LCT-O under these conditions has the same XRD pattern as that of LaCrO<sub>3</sub>; no new phases were observed. Thus, LCT-O is chemically stable under the conditions for H<sub>2</sub>S polishing.

The conductivities of the LCT-S and Li-LCT-S are shown in Fig. 5. The Li-LCT-S exhibited a higher conductivity than LCT-S. The conductivity of Li-LCT-S at 650 °C was about 0.02 S cm<sup>-1</sup>. The conductivities of both LCT-S and Li-LCT-S decreased as water was introduced into the inlet gas, indicating that the charge carriers are electrons that are formed owing to the removal of metal vacancies and oxygen ions [7]. The activation energy for conductivity of Li-LCT-S is about 50 kJ mol<sup>-1</sup>, while that for conductivity of LCT-S is about 93 kJ mol<sup>-1</sup>.

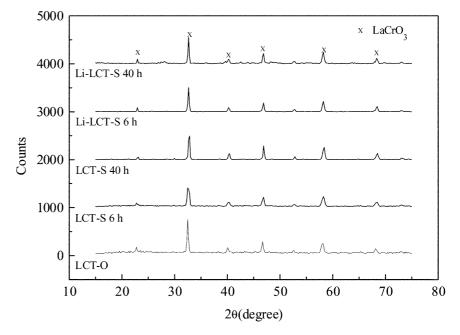
LCT-S and Li-LCT-S sintered at 900 °C have a uniform porous structure, as can be seen from the SEM images shown in Fig. 6a and b, indicating that the two materials have good thermal stability up to 900 °C. Thus, LCT-based sulfide and lithiated sulfide can be used as supporting substrates for electrochemical H<sub>2</sub>S polishing at temperatures up to 900 °C.

Y<sub>0.9</sub>Ca<sub>0.1</sub>FeO<sub>3</sub>

XRD patterns of CYF-O, CYF-S, and Li-CYF-S are shown in Fig. 7. It can be seen that CYF-O reacted with H<sub>2</sub>S and (Li<sub>0.62</sub>K<sub>0.38</sub>)<sub>2</sub>CO<sub>3</sub> to form new phases; in fact, the peaks corresponding to FeYO<sub>3</sub> can be identified from the XRD pattern of CYF-O in Fig. 7, which is significantly different from those of CYF-S and Li-CYF-S. The XRD patterns of CYF-S and Li-CYF-S are very similar, all including the phases of Y<sub>2</sub>O<sub>2</sub>S and FeS. These phases remained stable up to 40 h at 900 °C.

The conductivities of Li-CYF-S and CYF-S as measured at 650 °C are 15 and 3 S cm<sup>-1</sup>, respectively, as shown in Fig. 8. The conductivities are adequate for electrode applications. The conductivities of Li-CYF-S and CYF-S showed little dependency on temperature, which is significantly different from the characteristics of

Fig. 4 XRD patterns of LCT-O, LCT-S, and Li-LCT-S. The time adjacent to each spectrum represents the time that the sample was exposed to the gas mixture of 96% H<sub>2</sub> and 4% H<sub>2</sub>S at 900 °C



CYF-O under low oxygen partial pressures as reported by Kim and Yoo [10], indicating that the conduction mechanism in CYF-O is completely different from that in CYF-S. The former appears to be semiconducting, whereas the latter seems to be metallic. This is reasonable, considering the differences in phase structure between CYF-O and CYF-S.

Shown in Fig. 9 are the typical SEM views of assintered CYF-S and Li-CYF-S. Both samples are porous with a porosity of about 30%, implying that the melting points of the two materials are relatively high (>900 °C). The grain sizes of CYF-S and Li-CYF-S are not uniform.

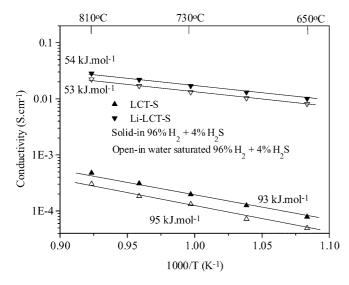


Fig. 5 Arrhenius plots of conductivities as measured in dry or humidified gas mixtures (96%  $H_2+4\%$   $H_2S$ ) of LCT-S and Li-LCT-S pellets sintered at 900 °C for 2 h. The number adjacent to each line represents the activation energy for electrical conductivity

SrCo<sub>0.8</sub>Fe<sub>0.2</sub>O<sub>3</sub>

SFC-O reacted readily with H<sub>2</sub>S and (Li<sub>0.62</sub>K<sub>0.38</sub>)<sub>2</sub>CO<sub>3</sub>, as evident from the XRD patterns of SFC-O, SFC-S, and Li-SFC-S shown in Fig. 10. The phases of these materials changed dramatically with the exposure time and the addition of lithium carbonate salts. During the process of preparing SFC-S and Li-SFC-S, some black volatile materials were observed, implying that some low-melting point phases were formed. However, SFC-S and Li-SFC-S samples had a porous structure with a porosity of 30% and 50%, respectively, as shown in Fig. 11. The conductivities of SFC-S and Li-SFC-S exhibit little dependence on temperature, as shown in Fig. 12. The conductivity of Li-SFC-S at 650 °C is about 200 S cm<sup>-1</sup>, which is excellent for electrode applications.

The highly porous microstructures and high conductivities of pretreated SFC-S and Li-SFC-S imply that a promising electrode based on the family of SFC-O may be obtained through tailoring the composition of the material.

#### **Conclusions**

The addition of lithium into the sulfides significantly increases the electrical conductivity but does not lead to a significant change in phase structure. Lithiated sulfide material derived from CYF, i.e., Li-CYF-S, exhibits high conductivity and good chemical stability in a gas

<sup>&</sup>lt;sup>1</sup>The stability and conductivity of materials are two important criteria for selecting suitable electrode materials. Yet, it is difficult to define the "good stability" of a material; it depends on the kinetics and thermodynamics of the reactions between electrode and reactants. A material that is practically stable under the operating conditions is not necessarily the one that is stable thermodynamically. In this paper, the stability refers to the relative stability of the materials investigated under the experimental conditions

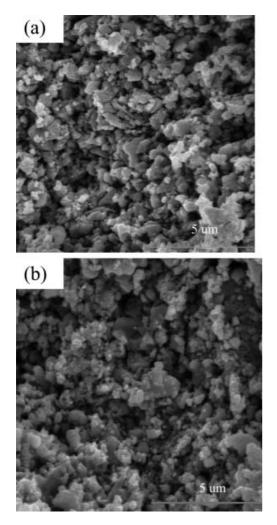
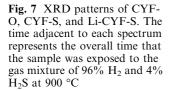
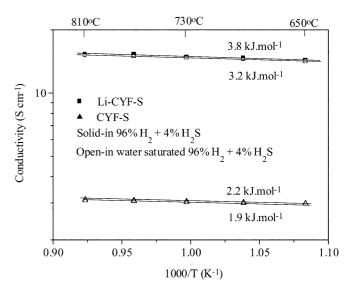


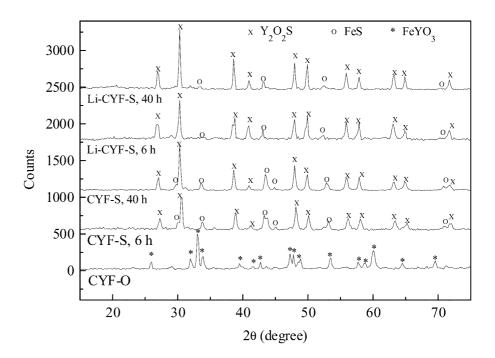
Fig. 6 SEM cross-sectional views of as-sintered pellets of a LCT-S and b Li-LSGC-S sintered at 900 °C for 2 h in 96%  $H_2+4\%$   $H_2S$ 





**Fig. 8** Arrhenius plots of conductivities as measured in dry or humidified gas mixtures (96%  $\rm H_2+4\%~H_2S$ ) of CYF-S and Li-CYF-S pellets sintered at 900 °C for 2 h. The number adjacent to each line represents the activation energy for electrical conductivity

mixture of 96% H<sub>2</sub> and 4% H<sub>2</sub>S at high temperatures, implying that it is a promising electrode material for electrochemical H<sub>2</sub>S polishing or for solid oxide fuel cells using H<sub>2</sub>S-containing fuels. However, the catalytic activity and long-term stability of these materials as electrodes are yet to be studied. Li-SFC-S also shows very high electrical conductivity, although the chemical composition of SFC-S and Li-SFC-S needs to be further modified to improve the stability. Both Li-LSGC-S and Li-LCT-S have relatively low electrical conductivity but have high structure and chemical stability; they may be used as substrate to support other active electrode materials or catalysts.



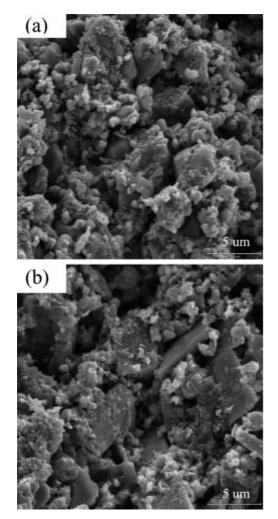


Fig. 9 SEM cross-sectional views of as-sintered pellets of a CYF-S and b Li-CYF-S sintered at 900 °C for 2 h in 96%  $\rm H_2+4\%~H_2S$ 

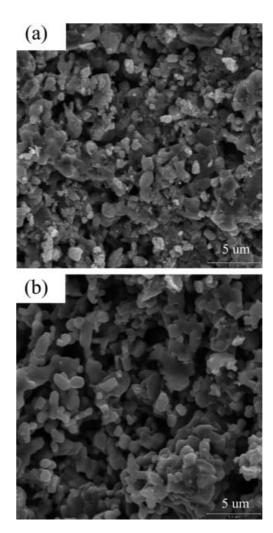


Fig. 11 SEM cross-sectional views of as-sintered pellets of a SFC-S and b Li-SFC-S sintered at 900 °C for 2 h in 96%  $\rm H_2+4\%~H_2S$ 

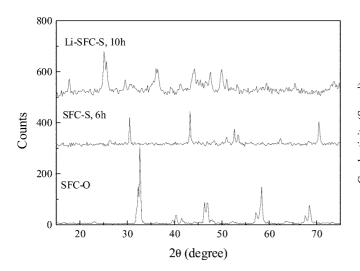


Fig. 10 XRD patterns of SFC-O, SFC-S, and Li-SFC-S. The time adjacent to each spectrum represents the overall time that the sample was exposed to the gas mixture of 96%  $\rm H_2$  and 4%  $\rm H_2S$  at 900  $^{\circ}C$ 

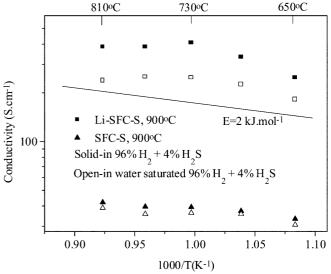


Fig. 12 Arrhenius plots of conductivities as measured in dry or humidified gas mixtures (96%  $H_2\!+\!4\%$   $H_2S)$  of SFC-S and Li-SFC-S pellets sintered at 900 °C for 2 h

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