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REVIEW PAPER

UDC 546.221.1:621.352:541.135:544.6

EVOLUTION OF FUEL CELLS POWERED BY H_2S-CONTAINING GASES

The development of the process and electrochemical materials for conversion of H_2S in a fuel cell to co-generate electrical power and benign products is outlined. While the thermodynamic basis for the process was clear, it was necessary to perform extensive research into the development of materials as catalysts and electrolytes, and to determine the optimal process and operating conditions. Through the use of composite anode catalysts and compatible new protonic electrolytes that are both chemically and thermally stable in the operating environment, we have achieved good and sustainable power densities. The only products are power, elemental sulfur and steam.

Key words: hydrogen sulfide; fuel cell; anode catalyst; electrolyte; cathode catalyst.

Each year large amounts of H_2S are generated worldwide, mostly as by-products from petroleum, natural gas and coal gasification industries. The adverse effects of H_2S on industrial processes, human health and the environment make it necessary to remove it from all effluent streams. It is anticipated that additional large quantities of H_2S will be produced due to the increase in utilization of high-sulfur crude oil and the expansion of coal gasification/liquefaction technology.

Many processes have been developed to remove and/or recover H_2S, including adsorption, absorption, hydrogen production and conversion to elemental sulfur via the two-step Claus process. Such H_2S processing units require high capital investments. Also, as the final products are of low commercial value, there is a high demand to find more economical and efficient hydrogen sulfide recovery methods. It is more desirable for hydrogen originating from H_2S to be electrochemically oxidized in a SOFC to generate electrical energy, leaving sulfur and water as exclusive anode reaction products. Thus, utilization of H_2S in solid oxide fuel cells provides a potentially economical and powerful option for concurrent chemical conversion and power (DC) generation.

The concept of parallel oxidation of one reagent and the reduction of the oxidant in separate compartments of an electrochemical cell has been known for over 100 years. When power is so generated, the system is a fuel cell [1,2]. The redox reactions are separated by an ion-conducting electrolyte membrane, and so the product at one side does not have the chance to react further with the reagent at the other side. Hence, through the selection of appropriate conditions for conversion of the feed, it is possible to convert the feed more selectively to desired products than is achievable when the oxidant and reductant are intimately mixed as was found, for example, in several attempts to convert hydrocarbons selectively to added value products [3]. Similar processes have been investigated for conversion of CH_4 to syngas and NO_x or SO_x to benign products [4]. Comparing our goals with the results from these earlier significant studies we concluded that it was necessary to develop a system for selective conversion of H_2S to elemental S, and probably would require a protonic membrane, reasoned as follows [5].

While the possibility that H_2S could be oxidized using a fuel cell (Eq. (1)) was recognized early, problems associated with managing the toxic and corrosive feed delayed the development of such systems. When the electrolyte membrane is oxide ion conducting the reactions are as shown in Eqs. (2) and (3), and when the electrolyte is proton conducting, the reactions are as shown in Eqs. (4) and (5). The first success was achieved using high temperature, oxide ion conducting solid oxide fuel cells (SOFC) [6-13].

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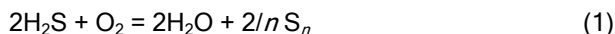Tel.: (780) 492-2232
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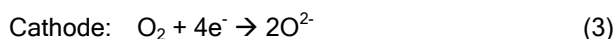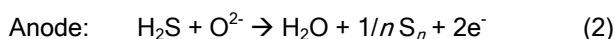Paper received: March 22, 2008.
Paper accepted: March 28, 2008.

However, none of these systems provided a sustainable high power output. In some cases the electrochemical materials themselves decomposed, and in others the anode catalyst rapidly deactivated. Thus, while these efforts were fruitful in demonstrating the feasibility of the process, none became the basis for commercial applications.
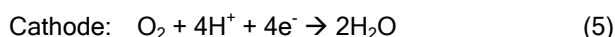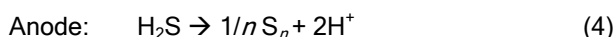
Overall reaction:

$$2H_2S + O_2 = 2H_2O + 2/n\,S_n \qquad (1)$$

$$\Delta G = 186 \text{ kJ mol}^{-1} \text{ (400 K)}$$

Oxide ion conducting:

Anode: $H_2S + O^{2-} \rightarrow H_2O + 1/n\,S_n + 2e^-$ (2)

Cathode: $O_2 + 4e^- \rightarrow 2O^{2-}$ (3)

Proton conducting:

Anode: $H_2S \rightarrow 1/n\,S_n + 2H^+$ (4)

Cathode: $O_2 + 4H^+ + 4e^- \rightarrow 2H_2O$ (5)

It was known that, when dissolved in an amine or other solvent, H_2S could be converted electrochemically at low temperatures to elemental sulfur, in either a fuel cell or electrolysis mode [14]. However, that process is difficult to operate, and generates colloidal sulfur.

The prior work with oxide ion conducting electrolytes showed that sulfur could form either elemental sulfur or sulfur oxides, primarily SO_2 [6-13]. Consequently, we concentrated our efforts towards the development of a protonic system, in which no SO_x would be generated as the sulfur species were never in contact with the oxidant. While conversion of H_2S at low temperatures in a protonic fuel cell is achievable [15], it is not desirable to produce solid sulfur in a gas phase fuel cell. Consequently, we first investigated the conversion of H_2S to elemental sulfur in the liquid sulfur region [16-18]. The concept and the process were proven, and could be operated over a range of temperatures using feeds that were either pure H_2S or mixtures of H_2S with diluent gases or light hydrocarbons [19]. Having shown the feasibility of the process, we showed that the system had good potential for the operation under a range of conditions [20].

While the results were of significant interest, the power densities achieved were insufficient for commercial operations and the stability of the system was uncertain. The data showed that there were several areas where improved performance was required. In particular, it was recognized that higher reaction rates were needed, and so the system should operate in the sulfur vapor region, at least over 450 °C and preferably higher. Thus, we needed several new materials. There were no stable protonic electrolytes that could sustain high conductivity at the desired temperatures. In contrast, oxide ion conductors required high temperatures, but these would then give a mixture of the products including SO_x. We needed stable catalysts for the activation of H_2S that could be strongly bonded to the electrolyte. We also needed cathode catalysts that were active and stable in the same temperature range. Thus, we undertook parallel and interactive investigations into each of these materials, and into their mutual compatibilities. We used a laboratory fuel having anode and cathode chambers comprising concentric feed and exhaust conduits bonded to each side of a membrane electrode assembly (MEA) (Figure 1).
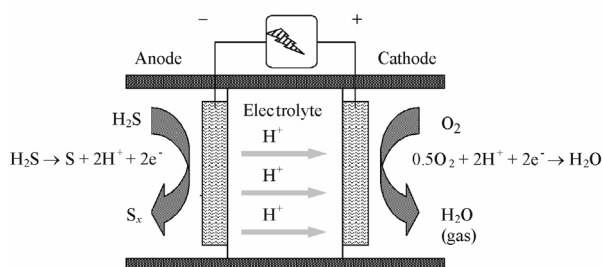

Figure 1. Schematic of the operation of proton conducting SOFC.

ANODE CATALYSTS

The initial fuel cell systems tested at both low and intermediate temperatures included metals, and especially noble metals, supported metal systems, oxides and sulfides [21]. We found that there was a propensity for metal catalysts to detach from the electrolyte, thus severely affecting the cell performance. The cause was determined to be a reversible formation of e.g. PtS. While a partial remedy was found through the use of an intermediate TiO_2 layer which "anchored" the Pt catalyst, the performance of the system was lower than that required for a commercial process [22]. Consequently, we then pursued the development of alternative catalysts, including metal sulfides and composite oxides.

A first series of sulfide catalysts was examined for the operation at temperatures in the range 450--800 °C [23]. The preferred range appeared to be 600--750 °C, the temperatures at which the product was highly volatile S_2. It was found that while NiS, FeS, CoS, and MoS_2, for example, were somewhat active alone, the activity and stability were greatly enhanced through three changes: (*i*) composite sulfides of two or more metals were more active than either alone; (*ii*) addition of about 5 wt % Ag enhanced the electronic conductivity significantly, and (*iii*) the addition of about 5 wt% of a porous oxide, and in particular YSZ,

enhanced mass transfer. The combination of these three factors improved the performance by more than an order of magnitude (Figure 2). Care was required, as Ag reversibly forms Ag_2S, which decomposes at high temperatures, and so the composite catalysts were pre-heated to the operating temperature before the introduction of H_2S [24-26]. The composite materials are highly active as they have very extensive triple phase boundaries, at which the reactants are activated to form electrons and ions which are conducted away separately [27].
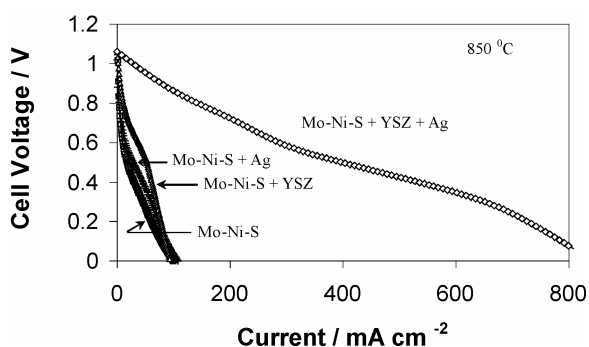

Figure 2. Performance enhancement through admixing Mo-Ni-S anode catalyst with 5 % each of Ag and YSZ.

The compositions and morphologies of the composite anode materials provide a good mass transfer, as demonstrated by the influence of the gas rate on their performance [28]. The magnitude of the change in OCV with temperature was consistent with calculated values based on thermal reaction equilibria (Eq. (6)), a reaction previously exploited for use in oxide ion conducting cells [29]. It was found that increasing either or both of air flow rate and H_2S flow rate im-

proved a current-voltage and power density performance. The results were consistent with improved gas diffusion in the cathode with increasing the air flow rate, and with both improved gas diffusion in the anode and the increased concentration of anodic electroactive species with increasing the H_2S flow rate.

$$H_2S \leftrightarrow H_2 + 1/n\,S_n \qquad (6)$$

The composition of the catalysts based on composite sulfides was examined in detail, and their stability and activity were demonstrated [30,31]. Curiously, when the catalyst was MoS_2 alone, the initial activity diminished with time, and this was found to be due to loss of the catalyst. While this behavior has been attributed to volatility of MoS_2, we later showed that was not the case as MoS_2 itself is not volatile. However, we found that even traces of O_2 present during the catalyst pre-treatment in the fuel cell convert MoS_2 to MoO_3, which is volatile. In contrast, when the catalyst comprises a composite sulfide of two metals, no Mo is lost through volatilization of MoO_3. Thus, the use of composite sulfides not only enhances the activity, it also enhances the stability (Figure 3) [32,33].

Systematic examination of a series of composite sulfide catalysts, AV_2S_4, determined that catalysts in which A was one of several transition elements had a superior activity when compared to any of the metal sulfides alone, or the prior benchmark Mo-Ni composite sulfide catalysts (Figure 4). The superior performance was attributable to the combination of high catalytic activity and low inherent anodic polarization resistance.
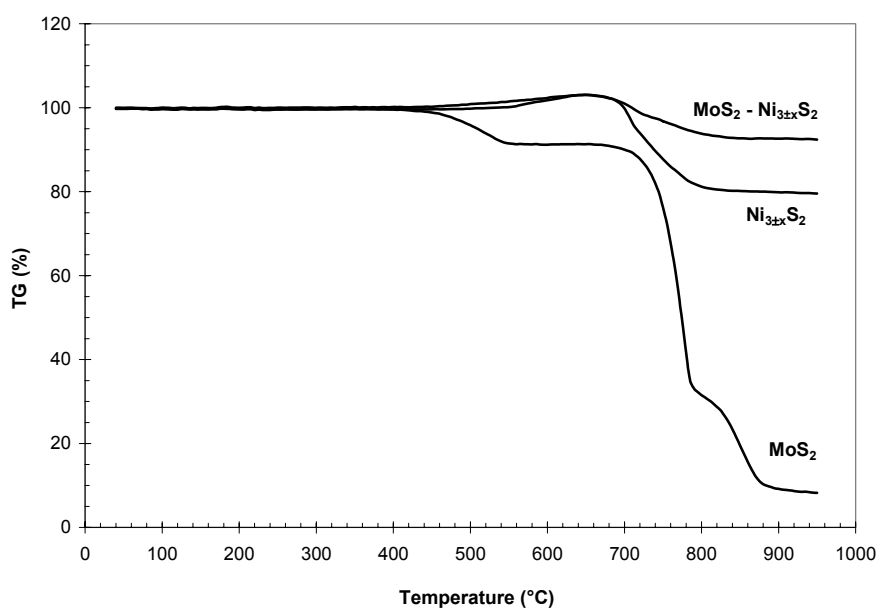

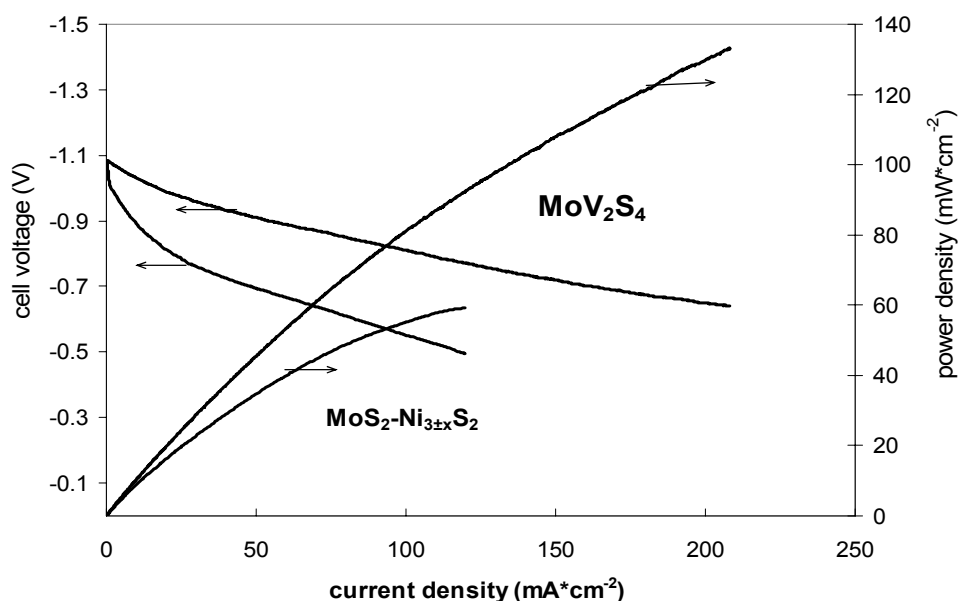Fig. 3. TGA analyses showing thermal stability of composite MoS_2-$Ni_{3\pm x}S_2$ anode catalysts.

Fig. 4. Comparison of the performance of MoV_2S_4 and MoS_2-$Ni_{3\pm x}S_2$ anode catalysts at 800 °C.

We determined the polarization resistances of a series of Mo, V and Cr ternary sulfide based materials of the general type AB_2S_4 (where A= Ni, Co, Fe, Mo, Cu and B = V, Mo, Cr). Those materials that were stable and had low resistances were then evaluated as potential anode materials for H_2S/air fuel cell applications. We found that the V-containing catalysts have a superior performance when compared with previously known MoS_2 and MoS_2-$Ni_{3\pm x}S_2$-Ag-YSZ anodes. The empirical trend for the polarization resistance obtained by using symmetrical cells and the relative activities for H_2S oxidation are parallel: MoV_2S_4 > > NiV_2S_4 > CrV_2S_4 > $CuCr_2S_4$ > MoS_2 > NiS-MoS_2-Ag- -YSZ > $CoMo_2S_4$ > $FeCr_2S_4$. The new anode materials are chemically, electrochemically and thermally stable during the operation of SOFC at 750-850 °C.

While the composite sulfide catalysts have proven to be useful in combination with different types of electrolyte, for example YSZ, cerates and ionic sulfates [34], and good power densities have been attained, their performance was limited. We recognized that we needed anode catalysts that were both more stable and had higher activity at high temperatures than the known systems. Consequently, in light of the high performance of the AV_2S_4 catalysts, we now have developed novel V-based catalysts comprising a mixture of $LaCrO_3$, a vanadium oxide and YSZ [35]. Advantageously, this family of anode catalysts is not poisoned by any of H_2S, elemental S, CO, CO_2, or hydrocarbons, and so can be used for the conversion of various feed streams containing H_2S, including industrial syngas (Figure 5) [36].
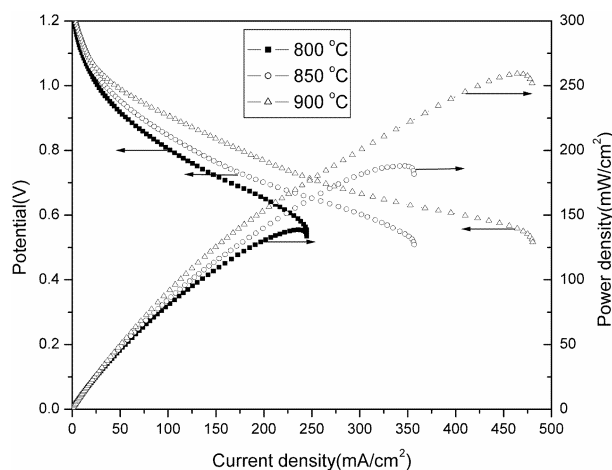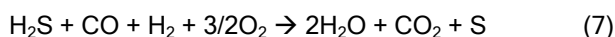

Figure 5. I-V and I-P performance of $LaCrO_3$-VO_x-YSZ in H_2S-containing syngas.

The new, highly active and stable anode catalyst $LaCrO_3$-VO_x-YSZ was developed specifically to determine whether we could operate a solid oxide fuel cell systems using impure hydrogen, and in particular industrial syngas derived from the conversion of coal or coke. Such raw syngas always contains H_2S, the majority of which must be removed for use with conventional catalysts. We found that the presence of CO in the anode feed enhanced fuel cell performance when compared to use of pure H_2 or H_2 containing 5000 ppm H_2S. Impedance spectra showed that the anode polarization resistance decreased in the presence of CO. High current density of 450 mA cm^{-2} at 0.6 V and maximum power density of 260 mW cm^{-2}

(at 450 mA cm^{-2}) was obtained at 1173 K. XRD and XPS analysis showed that the new catalyst was chemically stable in H_2S-rich syngas or hydrogen. However, when the CO concentration was high, slow carbon deposition compromised fuel cell performance during long tests at high temperatures.

When an oxide ion conductor is used, each of the components is oxidized at the anode to form H_2O, CO_2 and one of S_2, SO_x and a mixture thereof (Eq. (7)).

$$H_2S + CO + H_2 + 3/2O_2 \rightarrow 2H_2O + CO_2 + S \qquad (7)$$

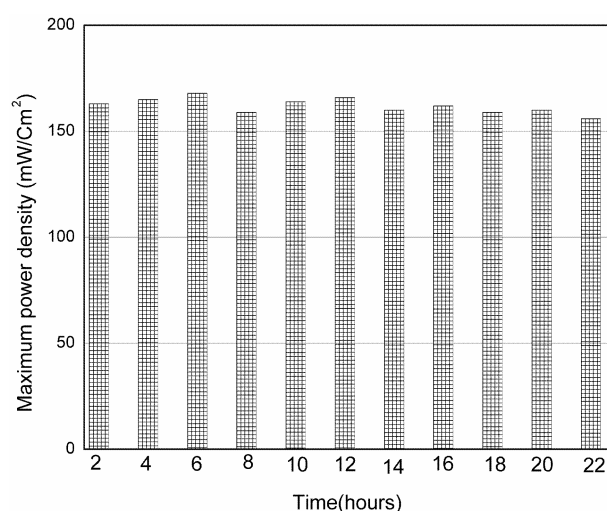The catalysts are stable under a variety of conditions, including the use in contaminated H_2 feed (Figure 6).

Figure 6. Performance of LaCrO$_3$-VO$_x$-YSZ anode catalyst as a function of time in H$_2$ + 5000 ppmv H$_2$S at 900 °C.

ELECTROLYTE

The electrolyte for a fuel cell must be highly conductive for the selected ions, chemically and thermally stable, and compatible with both the anode and the electrode.

Initially, it was not known whether there would be any benefit from the use of nano-sized particles in electrolyte membrane synthesis. We showed that the performances of nano-composite Li_2SO_4-Al_2O_3 membranes in H_2S-O_2 fuel cells were much superior to those prepared by using convention micro-particles (Figure 7) [37,38]. Thus, in all cases, we now compare the activities of membranes made from different size particles.

While the sulfate-alumina electrolytes are conductive, the proton number is low and so they are not useful for the present fuel cell applications [39,40]. Consequently, we have developed new protonic conductors, including several promising perovskites.
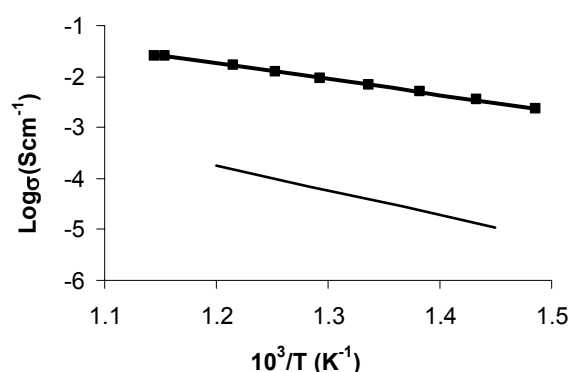

Figure 7. Superior ion conductivity of nano-composite vs. conventional Li$_2$SO$_4$-Al$_2$O$_3$ membranes.

It was well known that cerate perovskites have high protonic conductivity. However, they are unstable to decomposition and loss of the electronic structure required for conductivity. Further, they react with H_2S to form low conductivity sulfides. In contrast, zirconates are much more stable, but have low conductivity. Efforts elsewhere have examined the use of substituted perovskites. We found that Y-doped Ba(Ce,Zr)O$_3$ perovskites are the first protonic electrolytes proven to be chemically and thermally stable protonic conductors when used in solid oxide fuel cells operating at intermediate temperatures (450-700 °C) in hydrogen containing at least 500 ppm H_2S [41]. They are stable in fuel cells in which the feed is any one or combinations of H_2, CO and H_2S. They have high conductivity in all cases (e.g. Figure 8). The structures were characterized and the stability was confirmed using XRD, SEM and EDX. Membranes made from the BZY powders are stable proton conductors in H_2S-containing atmospheres, whereas undoped barium cerate has high proton conductivity but poor chemical stability. The resistance of BZY, measured by impedance spectroscopy, is strongly dependent on the temperature and the atmosphere.

In particular, the H_2S activation catalyst-electrolyte combinations are highly active and stable when used with H_2S-containing syngas (H_2 and CO with 5000 ppm H_2S). Thus this membrane assembly is very useful, as it allows the use of industrial syngas without extensive (and expensive) pre-conditioning, unlike the requirements for feeds for conventional fuel cells containing catalysts and electrolytes that are subject to poisoning by one or both of CO and H_2S.

CATHODE CATALYST

We have found that a variety of conventional cathode catalysts are compatible with each of the electrolytes we have used. Although there were issues related to the stability and the transfer of ions between

the materials during the use of some catalysts with sulfate-alumina composite electrolytes [42], no similar problems have arisen with the use of the perovskite electrolytes.
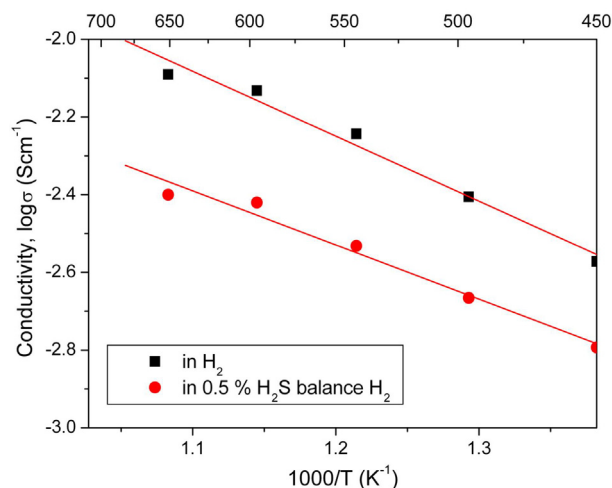

Figure 8. Bulk conductivity of BZY in H_2 and dilute H_2S as a function of temperature.

When different cathode catalysts (Pt, LSM and NiO) were tested with sulfate-alumina composite electrolytes, it was found that both Pt and LSM cathodes showed serious covering by electrolyte materials after its creeping at cell operating temperature [42]. In contrast, NiO appeared to be unaffected by electrolyte migration, and showed the highest cell performance with current density over 100 mA cm^{-2} and power density over 30 mW cm^{-2}. However, there was deleterious agglomeration of NiO particles after extended periods of operation.

CONCLUSIONS

The evolution of the H_2S-O_2 fuel cell process towards the present pre-pilot plant status required development of electrochemical materials for the use as anode and electrolyte systems that were chemically and thermally stable, and which were compatible with each other and the cathode. Preferred anode catalysts are composite metal sulfides, especially spinel and cubic AV_2S_4 catalysts (A = Ni, Mo, etc.). Electrolytes based on Y-doped Ba(Ce,Zr)O$_3$ perovskites are the first protonic electrolytes proven to be chemically and thermally stable protonic conductors when used in solid oxide fuel cells operating at intermediate temperatures in the presence of H_2S. Sulfate-alumina composite electrolytes are prone to migrate to cover Pt or LSM cathode catalysts, but not NiO. Thus, it is preferable to use protonic perovskite electrolytes. Preferably, the system operates at intermediate-to-high

temperatures (450-700 °C). The feed may be pure H_2S or any one of several industrial or resource streams containing H_2S, including H_2S streams, and relatively unrefined H_2 or syngas that would poison conventional systems. The attainable power density using pure H_2S or industrial syngas is sufficiently high for commercial applications. What is now required is testing using a scaled up unit over extended periods.

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