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Oxidation behavior of a Ni-Fe support in SOFC anode atmosphereNa Xu^{a,b,c}, Ming Chen^{b*}, Minfang Han^{a,c,d*},^a School of Chemical and Environmental Engineering, China University of Mining and Technology, Beijing 100083, China^b Department of Energy Conversion and Storage, Technical University of Denmark, Roskilde DK-4000, Denmark^c State Key Laboratory of Power Systems, Department of Thermal Engineering, Tsinghua University, Beijing 100084, China^d Tsinghua Innovation Center in Dongguan, Dongguan 523808, China

Abstract: In this work, we investigated the long-term oxidation behavior of a Ni-Fe (1:1 weight ratio) support for solid oxide fuel cell (SOFC) applications. Ni-Fe supports were obtained through tape casting, high temperature sintering and pre-reducing in 97% H₂/N₂ (9/91)-3% H₂O at 750 and 1000 °C, respectively. Then the Ni-Fe supports were exposed in simulated anode atmospheres of 97% H₂/N₂ (9/91)-3% H₂O and 75% H₂/N₂ (9/91)-25% H₂O for periods of up to 1000 h at 750 °C. The samples were examined for mass change, phase and chemical composition, and microstructure evolution during the annealing process. The Ni-Fe supports exposed to H₂/N₂-3% H₂O showed negligible oxidation, while those exposed to H₂/N₂-25% H₂O showed a 4-6% mass increase, due to the fact that a Fe-rich oxide scale was found on the surface of the Ni-Fe alloy particles. Room-temperature conductivity measurements showed that the supports annealed in the two atmospheres maintained sufficiently high conductivity. The results from the current work demonstrate that the porous Ni-Fe support can be well employed in SOFCs, especially metal-supported SOFCs.

Keywords: Ni-Fe support; Oxidation resistance; Metal-supported solid oxide fuel cell; Electrical conductivity

Solid oxide fuel cell (SOFC) technology has made significant progress over the past decades owing to improvements in materials, production techniques and other related technologies ^[1-3]. However, cost is still one of the main obstacles hindering the commercialization of SOFC technology ^[4]. Research into SOFC-based auxiliary power plant (APU) systems shows that a large part of the overall cost is the material cost of the SOFCs, which accounts for ~30 - 40% of the total system cost across the analyzed production volume. In addition, the high operation temperature of SOFCs imposes rigid requirements on stack components, such as the requirement of high-temperature alloys for interconnects (ICs) and thermal insulation materials,

which further increases the cost of SOFC technology^[5]. Therefore, it is paramount to reduce SOFC material costs.

State-of-the-art SOFCs are based on cermet supports for either the electrolyte or anode. Both have drawbacks of low mechanical strength, long start-up time, poor redox cycle capability and poor thermal stability. This hinders SOFCs from replacing widely used low-efficiency and high-polluting heat engines^[6]. Over the past decades, the SOFC configuration has evolved from a cathode-supported configuration to an electrolyte-supported configuration and further to the current anode-supported cell architecture. Recently, many efforts have been devoted to developing metal-supported SOFCs (MS-SOFCs), which are sometimes called third-generation SOFCs, to replace anode-supported configurations^[7-10]. Using metal as a support, the costly electrode and electrolyte layers can be made as thin as possible while ensuring their functionality, which results in an obvious cost reduction^[11]. MS-SOFCs also enable the use of matured metal welding processes as a sealing solution for SOFC stacks, simplifying the stack production and reducing costs. Last but not least, metal supports have decent thermal conductivity, which is expected to speed the start-up process and thus enable rapid thermal cycling of the SOFC stacks^[12].

Materials for MS-SOFC supports can be divided into two types: Ni-based and Fe-based materials^[13]. Ni shows great catalytic activity towards anodic reactions and high mechanical strength under SOFC working environments. Compared to Fe-based metal supports, Ni-based supports possess better electrical properties. However, the thermal expansion coefficient (TEC) of Ni is relatively high ($16.5 \times 10^{-6} \text{ K}^{-1}$), which causes difficulties during cell fabrication and operation. Furthermore, Ni also shows poor redox tolerance and is subject to performance degradation caused by carbon deposition and sulfur. All of the above factors limit the development of Ni-based metal supports for MS-SOFCs^[14-15]. Compared with Ni-based metal alloys, Fe-based alloys offer a wider range of applications as well as an attractive price. Crofer22 APU^[16], ZMG232^[17], and STS430^[18, 19] are commonly used as metal supports. However, Fe-based alloys are more easily oxidized in moderately to highly humidified anode atmospheres^[20], whereas Ni remains metallic in such environments.

Fe and Ni have complementary properties and a wide solubility range. Adding Fe into a Ni-based metal support will not only reduce the material cost but also decrease the TEC of the support. Ni-Fe alloys have attracted increased attention as metal support materials for MS-SOFCs^[21]. Usually, a porous Ni-Fe support is fabricated by mixing NiO and Fe₂O₃ and sintering the mixture in air, followed by reduction in H₂

^[22]. To obtain a TEC close to that of commonly used YSZ electrolyte material, a 1:1 mixture of Ni-Fe is typically used^[23]. Though MS-SOFCs have attracted wide interest, very few studies have been devoted to the oxidation behavior of SOFC metal supports under various anode atmospheres, such as H₂/H₂O or reformed gases^[24]. Though the effective oxygen partial pressure $p(\text{O}_2)$ on the anode side is rather low (between 10^{-20} and 10^{-8} atm), the porous metal support for SOFCs can still be easily oxidized due to the presence of water vapor^[25,26]. Sufficient oxidation resistance and electrical conductivity research of the porous metal support is thus essential for application. In this work, we investigated the oxidation behavior of a porous Ni-Fe alloy in diluted and humidified H₂ at 750 °C, simulating the anode compartment of MS-SOFCs. The evolution of the support microstructure and oxide scale formation was examined for oxidation periods of up to 1000 h. The samples were further evaluated in terms of their porosity (for gas transport) and electrical conductivity. The results of the current work are essential for the development of MS-SOFCs.

2. Experimental

Conventional tape-casting techniques were applied in the current work to produce the Ni-Fe metal support. According to a Ni:Fe mass ratio of 1:1, stoichiometric amounts of NiO and Fe₂O₃ powder (Huatsing Power, China) were weighed and mixed with zirconia ceramic grinding balls, solvents, pore former, binder, and plasticizer. The slurry was ball-milled for 48 h and then tape-casted into green tape on a casting machine. The green tape was punched into disks with a diameter of 20 mm and thickness of ~750 μm. The disks were also punched with a small hole (diameter of 3 mm) to allow the samples to be hung in the following oxidation tests. The disks were sintered at 1350 °C for 5 h. To reduce the support, the samples were placed in a tube furnace supplied with a gas mixture of 97% H₂/N₂ (9/91)-3% H₂O. For safety reasons, H₂ was diluted by N₂ with a ratio of H₂/N₂ = 9/91 to avoid any possible ignition. Two reduction profiles were employed: one at 750 °C for 10 h (denoted Sample A), the other at 1000 °C for 2 h (denoted Sample B). After reduction, the mass of Samples A and B reduced to 76.0wt% and 74.9wt% of original sample, respectively (Theoretically, the mass of the Ni-Fe porous support should be 74.0 wt %).

After reduction, the Ni-Fe porous support samples were hung in a cylinder-shaped alumina sample holder^[27]. By using this kind of sample holder, both sides of the sample could be equally exposed to the flowing gas. The alumina sample

holder was then placed in a horizontal tube furnace. For the oxidation tests, two kinds of atmospheres were used to simulate the environment in the anode compartment of a MS-SOFC: 97% H_2/N_2 (9/91)-3% H_2O (denoted H_2/N_2 -3% H_2O) and 75% H_2/N_2 (9/91)-25% H_2O (denoted H_2/N_2 -25% H_2O). The steam was produced by flowing the H_2/N_2 gas mixture to a water bath kept at a specific temperature (25 °C for 3% humidity and 70 °C for 25% humidity). The flow rate for the H_2/N_2 gas mixture was kept at 9 L/h. The samples were annealed at 750 °C for periods of up to 1000 h. A heating and cooling rate of 180 °C/h was employed. The oxidation tests were stopped at 100 h, 200 h, 300 h, 450 h, 600 h, 800 h and 1000 h, after which the samples were weighed.

At 0 h (i.e., after reduction), 100 h, 300 h, and 1000 h, one sample from each group was removed for post-test analysis. The samples were first examined by X-ray diffraction (XRD) using an automated Bruker D8 Advance spectrometer with Cu $\text{K}\alpha$ radiation. The XRD scan was carried out with a step size of 0.01° per 0.5 s over a 2θ range of 10 to 100°. The sample microstructure was examined using a Zeiss Merlin scanning electron microscope (SEM) equipped with an X-ray energy dispersive spectrometer (EDS). For the cross-sections, the samples were embedded in epoxy resin and polished by diamond abrasive. The samples were coated with carbon prior to imaging and EDS analysis. The porosity of the samples was obtained by SEM image analysis using the software ImageJ^[28, 29]. Finally, the conductivity of the Ni-Fe porous support samples was measured at room temperature using the Van der Pauw method as described previously^[30, 31].

3. Results and discussion

Fig. 1 (a) Calculated phase diagram of Fe-Ni-O₂ at 750 °C. The phase names are taken from FACTSAGE. The theoretical oxygen partial pressures of the two atmospheres used in the current work are indicated in the plot. (b) Calculated mass of the equilibrium phases for a mixture of 1 g Ni + 1 g Fe equilibrated at 750 °C as a function of $\log p(O_2)$.

Metal supports can be reduced at low oxygen partial pressure and oxidized again by increasing the oxygen partial pressure^[32]. Therefore, it is essential to explore the

equilibrium phase relations of the relevant systems. This was done in the current work by calculating the equilibrium phase diagram of Ni-Fe-O₂ using the FACTSAGE Thermochemical Software and Databases^[33]. A calculated phase diagram of Fe-Ni-O₂ at 750 °C is presented in Fig. 1(a), while Fig. 1(b) plot the mass of the equilibrium phases, for a mixture of 1 g Ni + 1 g Fe equilibrated at 750 °C as a function of log p(O₂). The phase names were taken directly from FACTSAGE. ASpinel and AMonoxide correspond to (Fe,Ni)₃O₄ and (Fe,Ni)O, respectively, while FCC_A1 and BCC_A2 correspond to the metallic phases. A red line corresponding to a Ni/Fe mass ratio of 1:1 is included in Fig. 1(a). In addition, the theoretical oxygen partial pressures for the two atmospheres employed for the oxidation tests in the current work are indicated in Fig. 1(a): H₂/N₂-3% H₂O (red dot) and H₂/N₂-25% H₂O (blue triangle) with p(O₂) = 10^{-21.398} atm and 10^{-19.022} atm, respectively.

As shown in Fig. 1a, the red dot is located in the FCC_A1 single-phase region, and the blue triangle is located in the FCC_A1+ASpinel region. Theoretically, Ni-Fe porous support should not be oxidized under H₂/N₂-3% H₂O atmosphere, but will partially oxidized to ASpinel using H₂/N₂-25% H₂O.

Fig. 1 (b) shows the equilibrium phase mass for Ni_{0.5}Fe_{0.5}O_x as a function of log p(O₂), assuming the Ni-Fe alloy contains 1 g of Ni and 1 g of Fe. From this figure, we can see when p(O₂) is below 10⁻²⁰ bar, Ni-Fe maintains the FCC_A1 phase. As the oxygen partial pressure increases, the metal phase is partially oxidized to oxide phases. In the p(O₂) range from 10⁻²⁰ to 10⁻¹⁶ bar, part of the metallic phase is oxidized to ASpinel. When p(O₂) is above 10⁻¹⁶ bar, the AMonoxide phase is formed. The metallic phase (FCC_A1) disappears entirely when p(O₂) is above 10⁻¹⁵ bar.

Fig. 2 Mass change of the Ni-Fe metal support annealed at 750 °C in H₂/N₂-3% H₂O and H₂/N₂

-25% H₂O for periods of up to 1000 h.

Fig. 2 shows the mass changes of the samples during long-term annealing, and the average mass gains were calculated from ~3-4 samples. The mass increases of the two samples under H₂/N₂-3% H₂O are negative because both Samples A and B were not fully reduced under H₂/N₂-3% H₂O either at 750 °C or 1000 °C, and the residual oxides were gradually reduced into the Ni-Fe alloy following annealing at 750 °C in H₂/N₂-3% H₂O. After annealing for 800 h, both Samples A and B stopped losing weight, and Sample A even showed a small increase from 800 h to 1000 h, suggesting slightly oxidation of the supports under 3% H₂O. The maximum mass loss of Sample A was ~2.76%, and that for Sample B was ~0.94%, which is consistent with the oxygen content calculated from the initial reduction; the minor differences are attributed to weighing error.

In contrast, continuous mass increases were observed for both samples when annealing in H₂/N₂-25% H₂O atmosphere at 750 °C. The supports experienced a rapid mass increase during the initial ~100 h, after which the increase slowed. The maximum mass increases of Samples A and B were 4.71% and 6.48% after 1000 h, respectively. Compared with Sample B, Sample A showed a smaller mass increase, which is attributed to the increased amount of residual oxide after the initial reduction, as described above.

Fig. 3 X-ray diffraction patterns of the porous Ni-Fe support annealed at 750 °C: (a) Sample A in H₂/N₂-3% H₂O, (b) Sample B in H₂/N₂-3% H₂O, (c) Sample A in H₂/N₂-25% H₂O, and (d) Sample B in H₂/N₂-25% H₂O.

Fig. 3 presents the XRD patterns of the as-reduced porous Ni-Fe alloy supports annealed in different simulated anode atmospheres at 750 °C. After the initial reduction, both Samples A and B indexed well with the Ni-Fe alloy phase (PDF No.

47-1405), suggesting that the supports were reduced at both 750 °C and 1000 °C, which is consistent with our previous results within the detection limit of X-ray diffraction^[32].

Fig. 3 (a) and (b) show the XRD patterns after annealing in 3% H₂O humidified H₂ for 100 h, 300 h and 1000 h. The XRD results show that the Ni-Fe support was stable without any detectable secondary phases up to 1000 h, suggesting that the effect of the oxygen partial pressure in 3% H₂O is minimal and can be neglected.

Fig. 3 (c) and (d) present the XRD patterns after annealing in the simulated anode atmosphere containing 25% H₂O. Impurity phases of iron oxide (Fe₃O₄, PDF No. 76-0955) were clearly detected. Moreover, the iron oxide peak intensity continuously increased with annealing time, which suggested that this anode atmosphere promoted the growth of oxide scale and confirmed the mass increase to be due to Fe₃O₄ growth caused by the high steam content in the ~25% H₂O gas. H₂O can dissociate into OH⁻ and H⁺, and the ions can be incorporated into oxide scale, which affects the defect structure of the oxides and promotes the inward diffusion of oxygen, increasing the outward flux of cations and finally leading to continuous oxidation^[34, 35].

Fig. 4 SEM cross-section images of the porous Ni-Fe support: Samples A and B (a), (b) after reduction, (c), (d) after 1000 h in H₂/N₂-3% H₂O, and (e), (f) after 1000 h in H₂/N₂-25% H₂O.

Table 1. Porosity of the porous Ni-Fe support annealed at 750 °C.

Support	0h	1000h H ₂ /N ₂ -3% H ₂ O	1000h H ₂ /N ₂ -25% H ₂ O
A	34.3 %	34.7 %	33.5 %
B	22.5 %	26.9 %	21.4 %

Fig. 4 (a), (c) and (b), (d) show the SEM images of Samples A and B before and after oxidation in H₂/N₂-3% H₂O for 1000 h. The morphology of the porous alloys showed no significant change, but the pores in the alloy seemed to be enlarged after 1000 h. This observation correlated with the fact that the mass of the support decreased and the alloy was further reduced after 1000 h indicates that the porosity of the metal support must have increased. To verify this conjecture, the software ImageJ was used to quantify the porosity of the samples. Ten SEM cross-section images with 1000X magnification were used to calculate the porosity of the samples. The results showed that Sample A had an initial porosity of 34.3%, and after annealing in H₂/N₂-3% H₂O for 1000 h, the porosity increased to 34.7%. The porosity of Sample A oxidized in H₂/N₂-25% H₂O should decrease. After examining ten images and taking the average, Fig. 4 (e) gives a value of 33.5%. Fig. 4 (b), (d) and (f) show a similar trend. The porosity of these three samples were also tested by the same method, and we obtained porosities 22.5%, 26.9%, and 21.4% for Sample B after reduction, after annealing in H₂/N₂-3% H₂O for 1000 h, and after annealing in H₂/N₂-25% H₂O for 1000 h, respectively.

Obvious morphological differences between the two Ni-Fe support samples were observed under the initial conditions, as shown in Fig. 4 (a) and (b). At the same calcination temperature but a different reduction temperature, Sample A has a larger porosity than Sample B, and Sample A is composed of particles with different grain sizes, while the particles in Sample B are sintered together homogeneously.

Fig. 5 High-magnification SEM images of the cross-section of Samples A (a) and B (b) after reduction and (c), (d) after annealing in H_2/N_2 -25% H_2O for 1000 h. For the area shown in (d), the EDS point analysis and elementary map are shown in (e)-(h).

To observe the oxidation behavior of the samples more clearly and to reconfirm the oxide components, the partially enlarged view of Fig. 4 (a), (b), (e) and (f) and the corresponding EDS mapping of Sample B annealed at H_2/N_2 -25% H_2O for 1000 h are presented in Fig. 5. Comparing (c) and (d) with (a) and (b), after oxidizing in H_2/N_2 -25% H_2O for 1000 h, oxide scale was clearly formed. Sample A-1000 h shows some dispersed oxide precipitates in the outer part of the particles, while Sample B-1000 h generated more uniform oxide scale, and the partially enlarged view demonstrates that the external oxide scale was formed along the Ni-Fe alloy grain surface. The EDS compositional analysis of (d) is shown in (e), (f), (g), and (h). Spots 1 and 2 were taken in the oxide and alloy regions to calculate the atomic fraction of

different elements. Three points were selected and averaged for each spot. Spot 1 in the oxide region was entirely composed of Fe and O, almost no Ni was observed, and the atomic ratio of Fe to O was 3:3.88. In combination with the XRD result, the oxide layers should be Fe_3O_4 . For Spot 2 in alloy region, Ni, Fe and O were present simultaneously. In this region, the oxygen content was very low, and the Ni content was higher than the Fe content, because Ni was not oxidized after oxidation and some Fe was precipitated from the alloy and formed oxides.

Fig. 6 Conductivity of the porous Ni-Fe support measured at 25 °C.

Van der Pauw DC conductivity measurements were used to test the electrical conductivity (σ) of the samples. The in-plane resistivity of all samples was tested, and the electrical conductivity results were obtained by calculation. The conductivities of Ni and Fe alone are 1.43×10^5 S/cm and 1.00×10^5 S/cm, respectively [36]. The alloyed samples have a lower electrical conductivity than the pure metals, and the values changed after oxidation. The electrical conductivity of Sample A was 4.61×10^3 S/cm. During the 1000 h H_2/N_2 -3% H_2O reduction process, σ continuously increased, and the conductivity increased to 6.36×10^3 S/cm, which was ascribed to reduction of the oxide components. When oxidized in H_2/N_2 -25% H_2O , σ continually decreased, and Sample A annealed in H_2/N_2 -25% H_2O for 1000 h had a conductivity of 2.85×10^3 S/cm, which is sufficient to meet the needs of a MS-SOFC support. In the reducing atmosphere of H_2/N_2 -3% H_2O , Sample B has a similar growth trend as Sample A in the first few hundred hours, and σ increased to 1.41×10^4 S/cm at 600 h, after which σ showed no obvious change. In the H_2/N_2 -25% H_2O atmosphere, the σ of Sample B decreased at first and then was maintained at approximately 1.22×10^4 S/cm.

Compared to Sample A, Sample B maintained a higher conductivity, which we believe is due to the higher conductivity of the NiO-Fe₂O₃ support reduced at 1000 °C before and after oxidation.

4. Conclusions

In this work, the oxidation resistance and electrical conductivity of a Ni-Fe MS-SOFC support were evaluated in two kinds of simulated anode atmospheres, H₂/N₂-3% H₂O and H₂/N₂-25% H₂O. After annealing at 750 °C in H₂/N₂-3% H₂O for up to 1000 h, Samples A and B were not oxidized, and the alloy maintained the Ni-Fe phase. After annealing in H₂/N₂-25% H₂O, the thermally grown oxide scale outside Ni-Fe particle was observed and proved to be Fe₃O₄. The presence of water vapor in the anode gas promoted the oxidation, which was enhanced at increased oxygen partial pressure. Although Fe₃O₄ was enriched on the surface of the Ni-Fe support and influenced its electrical conductivity, the alloys still offered sufficient conductivity for application as a MS-SOFC support.

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Highlights:

- Ni-Fe-O phase diagram was calculated through FACTSAGE Thermochemical Software and corresponding Databases
- Fe_3O_4 was enriched on surface of Ni-Fe support after exposed in simulated anode atmospheres of 75% H_2/N_2 (9/91)-25% H_2O
- Ni-Fe alloys offered sufficient conductivity for long term application as a MS-SOFC support.