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# Effect of CeO<sub>2</sub> on oxidation and electrical behaviors of ferritic stainless steel interconnects with Ni–Fe coatings

Peng Fei You <sup>a,b</sup>, Xue Zhang <sup>a,\*</sup>, Hai Liang Zhang <sup>a,b</sup>, Hui Jun Liu <sup>a</sup>,  
Chao Liu Zeng <sup>a</sup>

<sup>a</sup> Laboratory for Corrosion and Protection, Institute of Metal Research, Chinese Academy of Science, No. 62, Wencui Road, Shenyang, 110016, China

<sup>b</sup> School of Materials Science and Engineering, University of Science and Technology of China, No. 96, JinZhai Road Baohe District, Hefei, Anhui, 230026, China

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## ABSTRACT

Ferritic stainless steels are promising materials for application in interconnects of solid oxide fuel cells (SOFC). The present problems to be solved urgently for using ferritic stainless steels as interconnects are their rapid increase in electrical resistance and the cathode poisoning caused by evaporation of chromia. In the present study, the Ni–Fe and NiFe–CeO<sub>2</sub> alloy coatings have been electro-deposited onto 430 stainless steels (430SS). During oxidation at 800 °C in air, an outer dense NiFe<sub>2</sub>O<sub>4</sub> layer and an inner protective Cr<sub>2</sub>O<sub>3</sub> layer have thermally grown on the coated samples. The NiFe<sub>2</sub>O<sub>4</sub> layer retards the outward migration of chromium effectively. The addition of CeO<sub>2</sub> reduces the growth rate of Cr<sub>2</sub>O<sub>3</sub> and decreases the number of pores near the oxide scale/alloy interface. Moreover, a higher electrical conductivity has been achieved by the addition of CeO<sub>2</sub>.

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## Introduction

Solid oxide fuel cells (SOFC), the electrochemical devices that generate electricity directly from fossil fuel, have attracted a lot of interests due to their lower emissions and higher efficiency compared to other electro-chemical devices [1,2]. As an important component of SOFC, interconnects provide electrical connects between individual cells and separate oxidant gases (cathode) from fuels (anode) [3,4]. With the progress in

fabrication of SOFC, its operating temperature has been reduced to 600–800 °C from 1000 °C, which makes it possible for some cheap metallic materials to be used as interconnects. Interconnects have to be inert to materials of both cathode and anode, as well as be tolerant of both reducing and oxidizing atmospheres at high temperatures. To avoid excessive stress and fracture during thermal cycling, the coefficient of thermal expansion (CTE) of interconnects must match well with the other components of SOFC. Among the commercial oxidation-resistant alloys, Cr<sub>2</sub>O<sub>3</sub>-forming ferritic

\* Corresponding author.

E-mail address: [xuezhang@imr.ac.cn](mailto:xuezhang@imr.ac.cn) (X. Zhang).

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stainless steels are promising candidates for interconnects [5–7]. However, the evaporation of chromia at high temperatures into cathode would decrease the efficiency of cells, which impedes the application of  $\text{Cr}_2\text{O}_3$ -forming ferritic stainless steels in interconnects [8–11]. Several electrical conductive coatings have been developed to reduce the evaporation of chromia. These coatings include reactive element oxides [12,13],  $\text{MAlCrYO}$  (M represents a transition metal such as Co, Mn and/or Ti) [14], conductive perovskites [15,16], and spinels [17–21]. Among these coatings, the spinels such as Mn–Co and Mn–Cu spinel coatings are more attractive because they exhibit high electrical conductivity and could effectively reduce the evaporation rate of chromia. Some Fe-containing spinels exhibit the closest CTE values to ferritic stainless steels, and thus are also candidates for interconnect coatings. It is reported that the CTE of  $\text{NiFe}_2\text{O}_4$  spinel ( $10.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ ) is close to that of the ferritic stainless steels ( $11 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ ) [22]. Moreover, the electrical conductivity of  $\text{NiFe}_2\text{O}_4$  ( $0.26 \text{ S cm}^{-1}$ ) [22] is around 25 times larger than that of  $\text{Cr}_2\text{O}_3$  ( $1 \times 10^{-2} \text{ S cm}^{-1}$ ) [23]. Due to the fact that beneath the spinel coatings is commonly developed a  $\text{Cr}_2\text{O}_3$  scale which would dominate the area specific resistance (ASR) of interconnects, the  $\text{NiFe}_2\text{O}_4$  spinel is a promising coating material for metallic interconnects of SOFC. Several techniques have been developed to prepare spinel coatings, including screen print, chemical vapor deposition, plasma spray and electroplating a metal or alloy followed by oxidation treatment. For preparing  $\text{NiFe}_2\text{O}_4$  spinel coatings, a simple and economic method is electroplating a Ni–Fe alloy onto the substrate with a following oxidation treatment. Up to now, there have been some reports on the preparation of  $\text{NiFe}_2\text{O}_4$  spinel coatings by electrodeposition and subsequent oxidation treatment [24–27]. Liu et al. [24] prepared a  $\text{NiFe}_2\text{O}_4$  coating on an Ebrite alloy by electroplating a Ni–Fe alloy followed by oxidation at  $800 \text{ }^\circ\text{C}$  for 150 h. The obtained  $\text{NiFe}_2\text{O}_4$  coating could improve greatly the oxidation resistance of the alloy and decrease its ASR to around  $13 \text{ m}\Omega \text{ cm}^2$ , and is also expected to serve as an effective barrier to reduce the Cr evaporation and to limit the growth of  $\text{Cr}_2\text{O}_3$  scale. The  $\text{Cr}_2\text{O}_3$  scale grown on the coated alloy was  $1 \text{ }\mu\text{m}$  thick. However, many voids were also observed in the  $\text{NiFe}_2\text{O}_4$  coatings. Geng et al. has carried out a series of work to develop a  $(\text{Ni}, \text{Fe})_3\text{O}_4$  coating by electrodepositing a Ni–Fe alloy with a subsequent oxidation treatment at  $800 \text{ }^\circ\text{C}$  [25]. After oxidation for 3 weeks (504 h), the  $\text{Cr}_2\text{O}_3$  scale beneath this coating is  $2 \text{ }\mu\text{m}$  thick, and the ASR is about  $100 \text{ m}\Omega \text{ cm}^2$ . The authors further prepared a  $(\text{Ni}, \text{Fe})_3\text{O}_4/\text{NiO}$  coating by compositely electrodepositing a Ni– $\text{Fe}_2\text{O}_3$  alloy coating and then exposure in air at  $800 \text{ }^\circ\text{C}$  [26]. After oxidation of 4 weeks (672 h), the  $\text{Cr}_2\text{O}_3$  layer formed on the coated steel is around  $2 \text{ }\mu\text{m}$  thick, and the ASR is about  $60 \text{ m}\Omega \text{ cm}^2$ . The above two coatings could decrease effectively the ASR of the steels, but is ineffective in inhibiting the growth of  $\text{Cr}_2\text{O}_3$  scale. Thereby, Geng et al. attempted to develop a new  $(\text{Fe}, \text{Co}, \text{Ni})_3\text{O}_4$  coating by electroplating a ternary Fe–Co–Ni alloy coating followed by oxidation treatment at  $800 \text{ }^\circ\text{C}$  [27]. The coated steel forms a  $1 \text{ }\mu\text{m}$  thick  $\text{Cr}_2\text{O}_3$  layer and has an ASR of  $50 \text{ m}\Omega \text{ cm}^2$  after oxidation of 3 weeks (504 h). This coating could decrease the ASR of the ferritic stainless steels, and also retard the growth of  $\text{Cr}_2\text{O}_3$  scale. Clearly, the above Ni–Fe spinel coatings could inhibit effectively the outward diffusion

of Cr and are also expected to reduce the ASR of metallic interconnects at high temperatures, but their effectiveness in decreasing the growth rate of  $\text{Cr}_2\text{O}_3$  scale is still to be improved.

It is widely acknowledged that the addition of the rare earth element to stainless steels can reduce the growth rate of  $\text{Cr}_2\text{O}_3$  and the number of voids at scale/substrate interface [28–33]. Some investigations have indicated that the Ce-modified coatings or surfaces of interconnects could reduce ASR and improve oxidation-resistant performances effectively [34–36]. In this study, Ni–Fe and NiFe– $\text{CeO}_2$  alloy coatings have been prepared on ferritic stainless steels, followed by oxidation treatment to develop  $\text{NiFe}_2\text{O}_4$  and  $\text{NiFe}_2\text{O}_4\text{–CeO}_2$  coatings. The oxidation resistance and ASR of the  $\text{NiFe}_2\text{O}_4$  and  $\text{NiFe}_2\text{O}_4\text{–CeO}_2$  coated samples were also tested, with an attempt to reveal the effect of  $\text{CeO}_2$  on the coating performances.

## Experiment

### Materials

A commercial 430 ferritic stainless steel (430SS) with a nominal composition of Fe-17.0%, Cr-1.0%, Mn-1.0%, Si-0.12%, C-0.03%, S-0.04% P (mass percent) was used as substrate in this work. The coupons with a dimension of  $10 \text{ mm} \times 15 \text{ mm} \times 2 \text{ mm}$  were tailored with a wire cutting machine. The samples were mechanically grinded with up to 1000 grit SiC papers, followed by ultrasonically rinsing in deionized water, acetone and ethanol, respectively. Before electro-plating procedures, the coupons were pickled in 25% $\text{HCl}$ –5% $\text{HNO}_3$  solution for 10 s at room temperature.

### Preparation of the Ni–Fe and NiFe– $\text{CeO}_2$ alloy coatings

The electroplating solution was prepared by dissolving  $200 \text{ g L}^{-1}$   $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ,  $60 \text{ g L}^{-1}$   $\text{FeSO}_4 \cdot 6\text{H}_2\text{O}$ ,  $40 \text{ g L}^{-1}$   $\text{H}_3\text{BO}_3$ ,  $30 \text{ g L}^{-1}$   $\text{NaCl}$ ,  $30 \text{ g L}^{-1}$   $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ,  $0.4 \text{ g L}^{-1}$   $\text{C}_7\text{H}_5\text{O}_3\text{NS}$  and  $0.3 \text{ g L}^{-1}$   $\text{C}_{12}\text{H}_{25}\text{NaSO}_4$  in deionized water. The PH of the solution was adjusted to 3.5 with 20%  $\text{H}_2\text{SO}_4$ .

The Ni–Fe alloy coatings were galvanostatically electroplated onto motionless 430SS substrates at  $10 \text{ mA cm}^{-2}$  for 18 min at  $60 \pm 1 \text{ }^\circ\text{C}$  with the pure Ni ( $40 \text{ mm} \times 40 \text{ mm} \times 2 \text{ mm}$ ) and Fe ( $20 \text{ mm} \times 20 \text{ mm} \times 2 \text{ mm}$ ) sheets as counter electrodes. The NiFe– $\text{CeO}_2$  composite coatings were galvanostatically electroplated onto 430SS substrates using the prepared solution with the addition of  $12 \text{ g L}^{-1}$   $\text{CeO}_2$ . The particle sizes of the  $\text{CeO}_2$  powders are 20–50 nm. The solution was stirred by a magnetic stirrer with agitation rate of 1080 rpm. The electro-deposition was carried out at  $10 \text{ mA cm}^{-2}$  for 18 min at  $60 \pm 1 \text{ }^\circ\text{C}$  with the cathode (430SS) rotating at a speed of 2 rpm, using the pure Ni and Fe sheets as counter electrodes.

### Oxidation and characterization

The samples with Ni–Fe and NiFe– $\text{CeO}_2$  alloy coatings were pre-oxidized at  $800 \text{ }^\circ\text{C}$  in air for 2 and 10 h, respectively, to analyze the initial stage of oxidation. Further oxidation measurements of the samples were carried out at  $800 \text{ }^\circ\text{C}$  in air for

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