

# Dual-Environment Effects on the Oxidation of Metallic Interconnects

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**Metallic interconnects in solid oxide fuel cells are exposed to a dual environment: fuel on one side (i.e., H<sub>2</sub> gas) and oxidizer on the other side (i.e., air). It has been observed that the oxidation behavior of thin stainless steel sheet in air is changed by the presence of H<sub>2</sub> on the other side of the sheet. The resulting dual-environment scales are flaky and more friable than the single-environment scales. The H<sub>2</sub> disrupts the scale on the air side. A model to explain some of the effects of a dual environment is presented where hydrogen diffusing through the stainless steel sheet reacts with oxygen diffusing through the scale to form water vapor, which has sufficient vapor pressure to mechanically disrupt the scale. Experiments on preoxidized 316L stainless steel tubing exposed to air-air, H<sub>2</sub>-air, and H<sub>2</sub>-Ar environments are reported in support of the model.**

**Keywords** dual environment, hydrogen, interconnect, oxidation, solid oxide fuel cell, stainless steel

## 1. Introduction

Materials development into producing metallic interconnects for solid oxide fuel cells (SOFCs) has many challenges to overcome. One key challenge is that the interconnect must allow the transmission of electrical charge through the interior and through both surfaces. These surfaces are exposed on one side to an oxidizing atmosphere (such as air) and on the other side to a reducing atmosphere for the fuel (such as H<sub>2</sub>).

Prior investigations with stainless steels (Ref 1-4) have shown that oxidation behavior, and thus the ability to allow the transmission of electrical charge, is different when exposed to the air side of a dual environment compared with exposure to a single environment of air. Here a dual environment is defined as exposure of a relatively thin piece of metal to H<sub>2</sub> on one side and to air on the other side.

The observed changes from a single environment to a dual environment include a more porous, flaky, and thicker scale in the dual environment (Ref 1-4). One possible explanation for this behavior is that hydrogen diffuses through the metal and changes the point-defect chemistry of the oxide, which enhances the diffusion of Cr in chromia scales (Ref 2). Another is that the hydrogen that diffuses through the scale reacts with the less stable oxide components of the scale, such as iron oxide (Ref 1).

The mechanism proposed here is that hydrogen diffuses through the metal and reacts with oxygen in the scale to form water. As will be shown later, sufficient vapor pressure (steam) may be generated to mechanically form pores in the scale.

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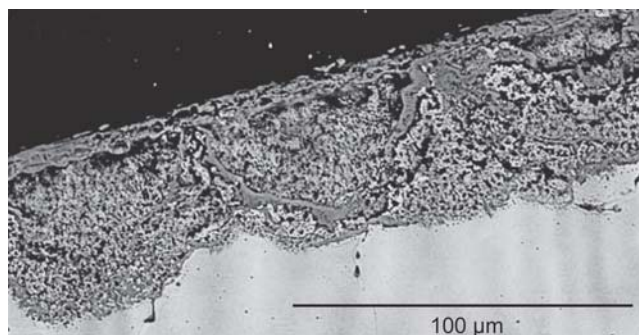
These pores then allow O<sub>2</sub> to diffuse as a gas through the scale and increase the oxidation rate. The flaky appearance of the scale could be explained by the disruption of the earliest-formed scale by steam pressure.

Some background into the formation of porous scales will be presented herein, along with calculations of steam pressures. A set of experiments that aimed to isolate the steam pressure mechanism from other possible hydrogen-scale interactions will also be presented.

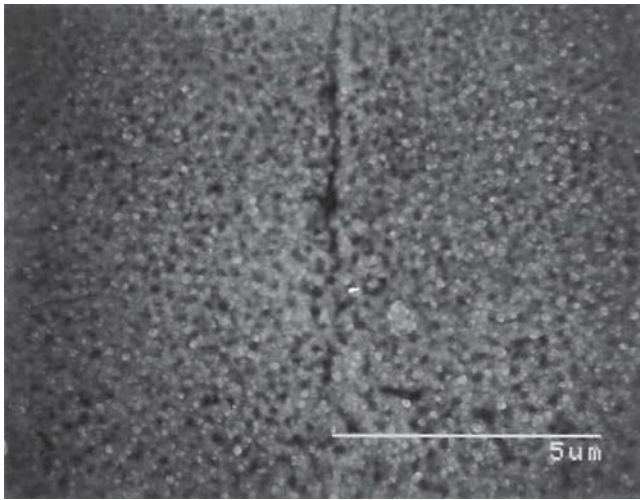
These experiments consisted of exposing preoxidized 316L stainless steel tubes to dual environments of H<sub>2</sub>-air and H<sub>2</sub>-Ar and to single environments of air-air and Ar-Ar. The lack of O<sub>2</sub> in the H<sub>2</sub>-Ar exposure would preclude steam generation as a scale-disruption mechanism. The dual environment of H<sub>2</sub>-Ar may show some effects of reactions of hydrogen with the less stable oxides in the scale (Ref 1) or with changes in the oxides that form (as compared with the Ar-Ar exposure) that result from increased Cr diffusion (Ref 2).

### 1.1 Porous Scales

The porous nature of the oxide scale formed in a dual environment can be seen in Fig. 1 for the air side of 316L exposed to a dual environment of moist H<sub>2</sub>-air for 96 h at 693 °C.



**Fig. 1** Porous scale formed on 316L from the air side of a dual-environment exposure of moist H<sub>2</sub>-air for 96 h at 693 °C.



**Fig. 2** Cross section of the porous hafnia scale formed at 1530 °C from the oxidation of HfC to HfO<sub>2</sub> and CO<sub>2</sub> (Ref 5)

This type of porosity is similar to the scales that can form when one of the oxidation products is a gas. One example is oxidation of HfC, which forms a porous HfO<sub>2</sub> scale and CO gas as reaction products at the carbide-oxide interface (Ref 5). The resulting scale is shown in Fig. 2. The partial pressure of CO that forms at the HfC-HfO<sub>2</sub> interface is such that pores form to allow the CO to escape. Subsequently, the CO reacts with O<sub>2</sub> from the gas phase to form CO<sub>2</sub> gas. This latter reaction occurs at what is termed the “flame front” within the scale (Ref 5). Oxidation rates are much higher for HfC than for Hf because the transport of oxygen can occur through the porous scale.

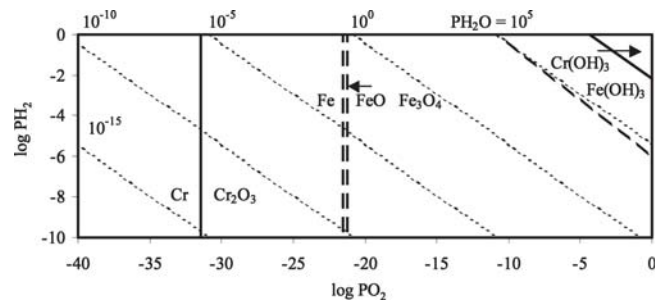
In a similar fashion, pores could be generated in the H<sub>2</sub>-air dual-environment scale from the formation of steam. The presence of the pores would then allow for rapid oxygen transport through the scale, resulting in more oxidation and thicker scales.

## 1.2 Steam Formation

The combination of the arrival of hydrogen through the metal and oxygen through the scale can result in formation of water. Water has a very low solubility in the scale, so it would be in the form of a gas, namely, steam.

Because chromia is very stable, the partial pressure of water is low at the metal scale interface, as shown in terms of the  $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$  ratio (Table 1). These calculations are estimates because the hydrogen or oxygen may be in ionic form and the activities of Cr and Cr<sub>2</sub>O<sub>3</sub> would not be equal to one. The very low  $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$  ratios are nonetheless illustrative that the reaction would not result in large steam pressures at the metal-scale interface.

However, there would exist a gradient of oxygen pressure or activity from 0.21 in the air to that of the  $P_{\text{O}_2}$  shown in Table 1. Assuming a linear gradient of oxygen partial pressure or activity, and a 1 μm thick scale, a partial pressure of oxygen of 0.0001 would occur ~0.5 nm (5 Å) from the metal. Using calculations similar to those done for Table 1, and assuming an oxygen partial pressure or activity of 0.0001, the  $P_{\text{H}_2\text{O}}/P_{\text{H}_2}$  ratios are much larger (Table 2). These large ratios suggest high steam pressures sufficient to mechanically alter the scale



**Fig. 3** Phase-stability diagram for the Cr-O-H (solid lines) and Fe-O-H (dashed lines) systems at 700 °C. Isobars of  $P_{\text{H}_2\text{O}}$  are shown as diagonal dotted lines.

**Table 1** Relative partial pressures for the formation of steam at the metal-scale interface

$T, ^\circ\text{C}$	$\Delta G_f \text{ Cr}_2\text{O}_3, \text{ kJ/mol}$	$P_{\text{O}_2}$	$\Delta G_f \text{ H}_2\text{O}, \text{ kJ/mol}$	$P_{\text{H}_2\text{O}}/P_{\text{H}_2}$
600	-903	9.6E-37	-200	8.6E-07
700	-878	3.8E-32	-194	5.2E-06
800	-853	2.1E-28	-189	2.2E-05
900	-828	2.7E-25	-187	1.1E-04

Based on thermodynamic data from Roine (Ref 6), on the reaction  $\frac{1}{2}\text{O}_2 + \text{H}_2 = \text{H}_2\text{O}$ , and with the activities of Cr and Cr<sub>2</sub>O<sub>3</sub> equal to 1

**Table 2** Relative partial pressures for the formation of steam within the scale near the metal-scale interface

$T, ^\circ\text{C}$	$P_{\text{O}_2}$	$\Delta G_f \text{ H}_2\text{O}, \text{ kJ/mol}$	$P_{\text{H}_2\text{O}}/P_{\text{H}_2}$
600	0.0001	-200	8.8E+09
700	0.0001	-194	2.7E+08
800	0.0001	-189	1.5E+07
900	0.0001	-187	2.2E+06

Based on thermodynamic data from Roine (Ref 6), on the reaction  $\frac{1}{2}\text{O}_2 + \text{H}_2 = \text{H}_2\text{O}$ , and with partial pressure or activity of oxygen equal to 0.0001

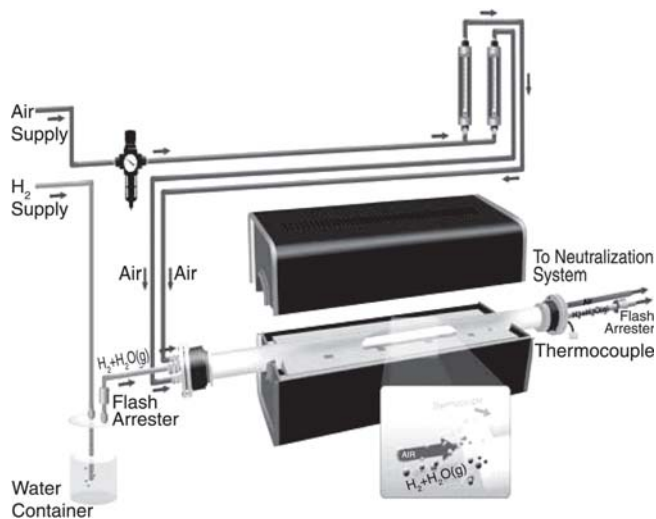
very near the metal interface. As the scale grows, it may sinter, which would result in fewer and larger pores. The image of such a scale would be in agreement with Fig. 1.

An alternative way to describe the possible steam pressures is with a stability diagram, as shown in Fig. 3. Figure 3 demonstrates the Cr-O-H and Fe-O-H systems at 700 °C in terms of  $\log P_{\text{H}_2}$  and  $\log P_{\text{O}_2}$ . Isobars of  $P_{\text{H}_2\text{O}}$ , the diagonal dotted lines, show partial pressures above 1, necessary for void formation, in the upper right-hand part of the diagram. For a  $P_{\text{O}_2}$  of  $10^{-4}$ , a  $P_{\text{H}_2}$  of  $\sim 10^{-8}$  is required for a  $P_{\text{H}_2\text{O}}$  of 1.

## 2. Experimental Procedures

Overall, the experiments consisted of exposing preoxidized 316L stainless steel tubes to dual environments of H<sub>2</sub>-air and H<sub>2</sub>-Ar and single environments of air-air and Ar-Ar. The resulting scales on the outer diameter of the tubes were then examined.

The 316L tubes (ID, 7.76 mm; OD, 9.32 mm) were preoxidized in dry air at 700 °C for 100 h. Both outside and inside diameters were preoxidized. With the exception of degreasing



**Fig. 4** Dual-environment test rig for tubular samples. The experiments described herein were done with dry H<sub>2</sub> (not bubbled through the water chamber).

Preoxidized, then H<sub>2</sub>-air



Preoxidized, then air-air



Preoxidized, then H<sub>2</sub>-Ar



Preoxidized, then Ar-Ar

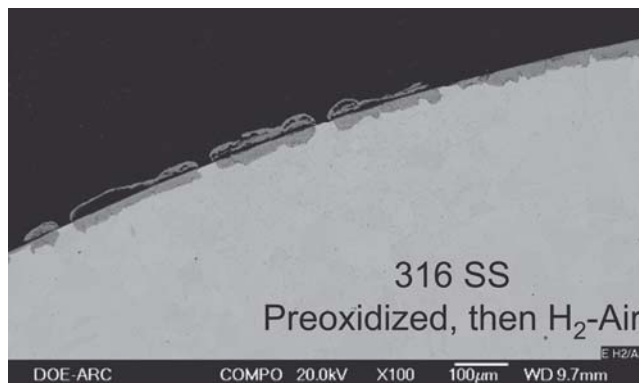


**Fig. 5** Outer-diameter scales after 100 h exposures at 700 °C

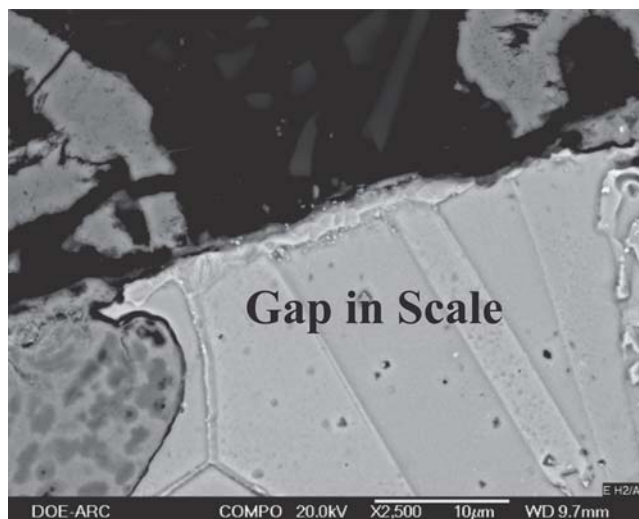
with methanol, the tubes were exposed in the “as-received” condition.

The preoxidized tubes were then exposed at 700 °C for 100 h in one of four conditions: H<sub>2</sub>-air, H<sub>2</sub>-Ar, air, and Ar. The dual-environment conditions (H<sub>2</sub>-air and H<sub>2</sub>-Ar) were always with the H<sub>2</sub> in the inside of the 316L tubes. The apparatus for the dual-environment experiments is shown in Fig. 4.

The single-environment conditions were used during the same furnace runs, but with 3 in. (7.6 cm) long tube pieces placed inside the furnace chamber. The furnace heat-ups and



**Fig. 6** Backscattered electron image of preoxidized 316L exposed to a dual environment of H<sub>2</sub>-air for 100 h at 700 °C. Shown is the outside diameter (the air exposure).



**Fig. 7** Backscattered electron image of preoxidized 316L exposed to a dual environment of H<sub>2</sub>-air for 100 h at 700 °C. Shown is the outside diameter (the air exposure) at the edge of a gap in the scale (Fig. 6).

cool-downs were done at 225 °C/h with N<sub>2</sub> used as the purge gas. The gas velocity inside the 316L tubes was approximately 430 cm/min. The gas velocity outside the 316L tubes was approximately 5 cm/min.

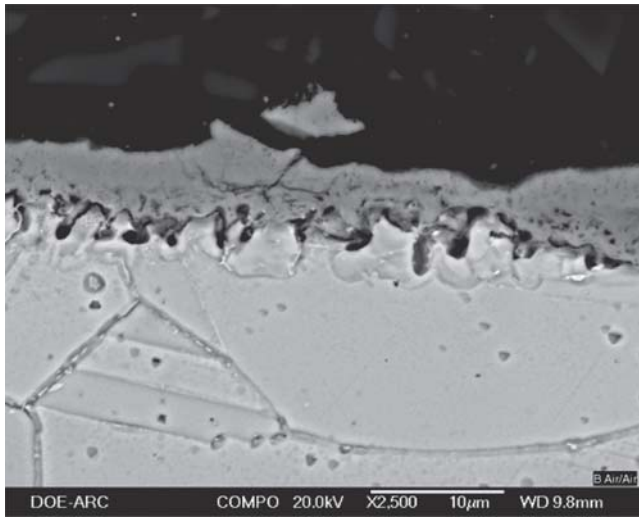
Post-exposure examinations consisted of visual examination of the outside diameter surfaces of the tubes, microscopy (light and scanning electron) of cross sections, elemental analyses of cross sections, and x-ray diffraction (XRD) of scales.

### 3. Results

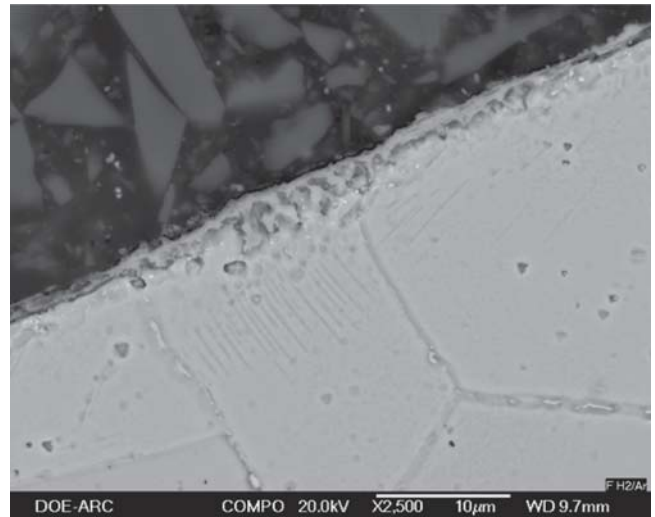
Figure 5 shows the outside diameter scales of the tubes. The dual-environment effect described earlier (Ref 1-4) can be seen by comparing H<sub>2</sub>-air with air-air. The H<sub>2</sub>-air shows the flaky scale characteristic of dual environments (Ref 1-4). There was little difference between the other scales shown in Fig. 5.

The discontinuous nature of the scale in the H<sub>2</sub>-air dual-environment scale can be seen in Fig. 6. The inner scale was a Cr-Fe-Ni oxide; the outer scale was Fe<sub>3</sub>O<sub>4</sub>. The outer scale is the flaky scale seen in the top of Fig. 5.

Higher magnification images of each of the exposures are shown in Fig. 7-10. Figure 7 is the H<sub>2</sub>-air dual environment



**Fig. 8** Backscattered electron image of preoxidized 316L exposed to a single environment of air-air for 100 h at 700 °C. Shown is the outside diameter (the air exposure).



**Fig. 9** Backscattered electron image of preoxidized 316L exposed to a dual environment of H<sub>2</sub> Ar for 100 h at 700 °C. Shown is the outside diameter (the air exposure).

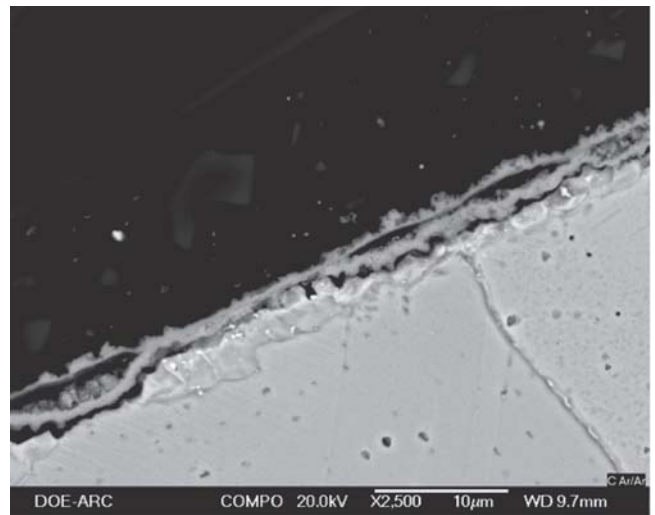
**Table 3 Results from XRD and elemental analyses for each of the four exposure types**

No elemental analyses are given for regions not observed in Fig. 6-10.

	H <sub>2</sub> /air	Air/air	H <sub>2</sub> /Ar	Ar/Ar
Figures	6, 7	8	9	10
XRD	FeCr <sub>2</sub> O <sub>4</sub> Fe <sub>2</sub> O <sub>3</sub>	FeCr <sub>2</sub> O <sub>4</sub> Cr <sub>2</sub> O <sub>3</sub>	FeCr <sub>2</sub> O <sub>4</sub> Cr <sub>2</sub> O <sub>3</sub>	FeCr <sub>2</sub> O <sub>4</sub> Cr <sub>2</sub> O <sub>3</sub>
Outer oxide elemental analysis, at.%				
O	57	56	...	60
Fe	42	40	...	15
Cr	0.6	1.8	...	18
Ni	...	0.7	...	0.1
Mo	...	0.2	...	0.2
Mn	0.2	0.9	...	3
Si	0.1	0.3	...	2
Inner oxide elemental analysis, at.%				
O	50	61	55	60
Fe	21	13	17	12
Cr	19	20	20	22
Ni	7	3	3	1.4
Mo	1.2	0.7	0.5	0.7
Mn	1.5	0.5	1.0	1.4
Si	0.9	1.0	1.9	1.3
Inner oxide elemental analysis (darker spots), at.%				
O	51	...	...	...
Fe	19	...	...	...
Cr	21	...	...	...
Ni	5	...	...	...
Mo	1.3	...	...	...
Mn	1.6	...	...	...
Si	1.1	...	...	...

near a gap in the scale (as seen in Fig. 6). X-ray diffraction and elemental analyses (Table 3) show the inner scale to be Cr-Fe-Ni oxide with a spinel structure and the outer scale to be Fe<sub>2</sub>O<sub>3</sub>.

Figure 8 shows a sample reacted in the air-air single environment. Table 3 shows that the outer part of the scale is Fe oxide. However, the XRD results are more consistent with it being Fe<sub>3</sub>O<sub>4</sub>. The inner oxide is Cr<sub>2</sub>O<sub>3</sub> and a Cr-Fe-Ni spinel. Thus the scale is much different than in Fig. 7, as it is much thicker, continuous, and contains no Fe<sub>2</sub>O<sub>3</sub> in the outer oxide.



**Fig. 10** Backscattered electron image of preoxidized 316L exposed to a single environment of Ar-Ar for 100 h at 700 °C. Shown is the outside diameter (the air exposure).

Figure 9 shows a sample reacted in the H<sub>2</sub>-Ar dual environment. The scale is much different than those found in Fig. 7 and 8. The scale is much thinner and consists of Cr<sub>2</sub>O<sub>3</sub> and a Cr-Fe-Ni spinel (Table 3). The thinness of the scale, as compared with Fig. 8, is not surprising; it was exposed to air at temperature for half of the time.

Figure 10 is the Ar single environment. As in Fig. 9, the scale is Cr<sub>2</sub>O<sub>3</sub> and a Cr-Fe-Ni spinel (Table 3). The oxide is partially detached from the metal and in general appears less protective than in Fig. 8. The separated part of the oxide scale is essentially the same as the attached part of the scale.

## 4. Discussion

### 4.1 Hydrogen Attack of Less Stable Oxides

Examining Fig. 5-8 for dual-environment mechanisms, one finds little evidence for hydrogen attack of less-stable oxides

within the scale (Ref 1). If this were the case, then Fig. 7 would show a reaction zone, and the scale would probably look less protective than in Fig. 8.

Figure 3 shows that for Cr-O-H and Fe-O-H systems, the formation of  $\text{Fe}(\text{OH})_3$  and  $\text{Cr}(\text{OH})_3$  could be possible at very high  $P_{\text{O}_2}$  and  $P_{\text{H}_2}$  conditions. However, XRD results revealed neither  $\text{Fe}(\text{OH})_3$  nor  $\text{Cr}(\text{OH})_3$ .

#### 4.2 Point Defect Modification of the Scale

The possibility that the dual-environment effect is due to protons modifying the oxide to allow for faster Cr or Fe diffusion through the scale (Ref 2) is more difficult to examine. This is because a lack of oxygen would also be expected to modify the point defects in the oxide scale.

For chromia scales, Kofstad (Ref 7) used electrical conductivity and diffusion studies to describe the point-defect structure in terms of oxygen activity:

The predominant point defects with very low oxygen activity, such as near the Cr-Cr<sub>2</sub>O<sub>3</sub> boundary, are chromium interstitials with an effective charge of +3,  $\text{Cr}_i^{3\cdot}$ . Oxygen vacancies,  $\text{V}_{\text{O}}^{2\cdot}$ , with an effective charge of +2, may be important minority defects. Under these conditions, chromia is an n-type semiconductor, so the electroneutrality condition (ENC) for chromium interstitials is  $3[\text{Cr}_i^{3\cdot}] = n$ , where  $n$  is the concentration of electrons.

At near atmospheric oxygen pressures, the predominant point defects are oxygen interstitials with an effective charge of -2,  $\text{O}_i^{2\cdot}$ , and chromium vacancies with an effective charge of -3,  $\text{V}_{\text{Cr}}^{3\cdot}$ . Under these conditions, chromia is a p-type semiconductor, so the ENC for chromium vacancies is  $3[\text{V}_{\text{Cr}}^{3\cdot}] = p$ , where  $p$  is the concentration of electron holes.

At intermediate oxygen activities the predominate point defects are stoichiometric structures, such as Frenkel ( $\text{Cr}_i^{3\cdot}$  and  $\text{V}_{\text{Cr}}^{3\cdot}$ ) or Schottky ( $\text{V}_{\text{Cr}}^{3\cdot}$  and  $\text{V}_{\text{O}}^{2\cdot}$ ) defect pairs. The ENC equates the concentration of each defect within the defect pair.

In terms of hydrogen changing the defect structure, the introduced defects could be described either as hydrogen interstitials,  $\text{H}_i^{\cdot}$ , or as OH complexes on oxygen sites,  $(\text{OH})_{\text{O}}^{\cdot}$ . Either case introduces a doping effect with defects having an effective charge of +1. For low oxygen activities, the ENC with doping could become  $3[\text{Cr}_i^{3\cdot}] + [(\text{OH})_{\text{O}}^{\cdot}] = n$ . This would tend to decrease the concentration of chromium interstitials and thus decrease the diffusion of Cr through this part of the scale. Conversely, for high oxygen activities, the ENC could become  $3[\text{V}_{\text{Cr}}^{3\cdot}] = p + [(\text{OH})_{\text{O}}^{\cdot}]$ . This would tend to increase the concentration of chromium vacancies and thus increase the diffusion of Cr through this part of the scale.

For chromia scales, then, one could conclude that an increase in oxidation rate in dual environments from a modification of the point-defect structure increasing the diffusion of chromium could be possible in the outer part of the scale with higher oxygen activities.

However, the situation described by point defects is confusing. Kofstad (Ref 7) has compared diffusion study results at oxygen activities found at the Cr-Cr<sub>2</sub>O<sub>3</sub> interface and has found higher diffusion rates for when oxygen activities were controlled with H<sub>2</sub>+H<sub>2</sub>O gas mixtures (Ref 8) than with CO+CO<sub>2</sub> gas mixtures (Ref 9). Thus, a simple doping model, and its effects on the ENC, may not be sufficient to describe the situation, even in the relatively simple case of chromia scales, let alone the case of multilayered Cr-Fe oxide scales.

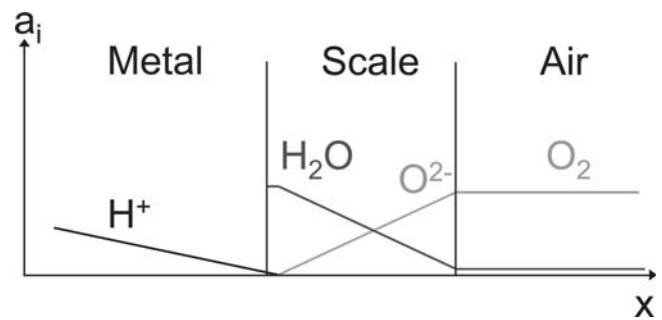


Fig. 11 Schematic of activity profiles during H<sub>2</sub>O formation prior to scale disruption. Oxygen transport through the scale is by point defects.

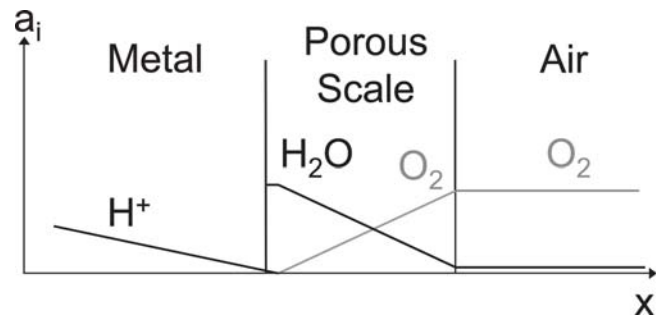


Fig. 12 Schematic of activity profiles during H<sub>2</sub>O formation after pore formation. Oxygen transport through the scale is by gaseous diffusion.

#### 4.3 Steam Pressure Effects

As described earlier under steam formation, H<sub>2</sub>O would be expected to form within the scale, albeit close to the metal-scale interface. When oxygen transport is by point defects within the scale, a picture of the activity profiles might resemble Fig. 11. The activity (or partial pressure) of H<sub>2</sub>O could be much higher than shown. When the partial pressure of H<sub>2</sub>O becomes high enough, pores may form to allow steam to escape (Fig. 12). The pores would also allow oxygen to diffuse into the scale as a gas.

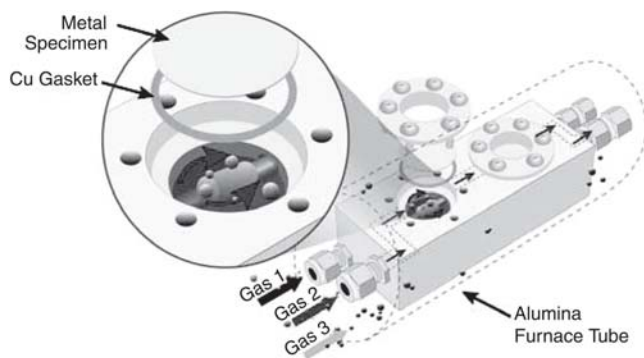
#### 4.4 Water Effects

In addition to the physical effects of high steam pressure, simply the presence of water within the scale can change the nature of the oxide scale and the kinetics of scale formation. It is well established that oxidation of steel and stainless steel in moist air, as opposed to dry air, can lead to faster oxidation, less protective scales, and breakaway oxidation (Ref 7).

For dual-environment exposures of Crofer22 APU, Yang et al. (Ref 10) have found that the scale in the air side of 97% H<sub>2</sub> + 3% H<sub>2</sub>O/air + 3% H<sub>2</sub>O contained hematite nodules, while no hematite was found in the scale in the air side of 97% H<sub>2</sub> + 3% H<sub>2</sub>O/air + 1% H<sub>2</sub>O. Thus, increased water vapor on the air side can also modify the scale morphology.

#### 4.5 Future Work

The experiments and discussion above were for 316L in a dual environment. Currently, a dual-environment apparatus is being used to test flat samples of more relevant SOFC metallic



**Fig. 13** Apparatus for testing flat samples in a dual environment. Specimen is sealed between the two environments with a copper gasket.

interconnect alloys (Fig. 13). Alloys being examined include Alloy 230, Crofer 22 APU, J1, and J5. Alloys J1 (Ni-18Mo-12Cr-1.1Ti-0.9Al) and J5 (Ni-22.5Mo-12.5Cr-1Ti-0.1Al-0.5Mn) (Ref 11) are nickel-based alloys designed for low coefficients of thermal expansion, a useful characteristic for conforming to SOFC seals. Alloy J1 is based on Mitsubishi alloy LTES700 (Ref 12).

One difference that can be expected for nickel alloys in dual environments is that there should be less of an effect than for iron alloys because nickel has a lower hydrogen permeability than does iron (Ref 13). At 700 °C, the permeability of hydrogen in pure nickel is  $\sim 5 \times 10^{-5}$  cm<sup>2</sup>/s; pure iron is  $\sim 3 \times 10^{-4}$  cm<sup>2</sup>/s (Ref 13).

In terms of Fig. 3, if the lower permeability of hydrogen in nickel-base alloys drops the  $P_{H_2}$  below that required for a  $P_{H_2O}$  above 1, then sufficient steam pressure would not be expected to form pores.

## 5. Summary

Dual environments (H<sub>2</sub> on one side and air on the other side) in SOFC systems can degrade the air-side scale compared with that formed in a single environment of air. The scale becomes thicker and more porous, and it tends to delaminate in flakes. All of these differences will degrade the current transfer ability that is required of a SOFC interconnect.

A mechanism to explain some of the effects of a dual-environment effect was presented that postulates the reaction of hydrogen (permeated across the metal) with oxygen (diffused

through the scale) to form water in the scale very close to the metal interface. The degradation (pores and delamination) of the scale then occurs due to high steam pressure close to the metal-scale interface. The presence of pores allows for faster diffusion of O<sub>2</sub> gas through the scale, resulting in thicker scales.

Experiments were conducted on preoxidized 316L stainless steel tubes in dual environments of H<sub>2</sub>-air and H<sub>2</sub>-Ar and single environments of air-air and Ar-Ar. The dual-environment effect was present in the H<sub>2</sub>-air exposure. No evidence was found for detrimental hydrogen effects in the H<sub>2</sub>-Ar dual-environment exposure.

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