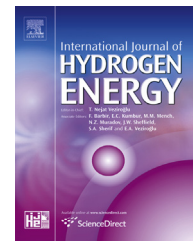




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# Dual atmosphere study of the K41X stainless steel for interconnect application in high temperature water vapour electrolysis

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## ARTICLE INFO

### Article history:

Received 11 July 2014

Received in revised form

1 December 2014

Accepted 20 January 2015

Available online 21 February 2015

### Keywords:

High temperature electrolysis

Interconnect

Dual atmosphere

Corrosion behaviour

Electrical current influence

## ABSTRACT

High temperature water vapour electrolysis (HTE) is one of the most efficient technologies for mass hydrogen production. A major technical difficulty related to high temperature water vapour electrolysis is the development of interconnects working efficiently for a long period. Working temperature of 800 °C enables the use of metallic materials as interconnects. High temperature corrosion behaviour and electrical conductivity of a commercial stainless steel, K41X (AISI 441), were tested in HTE dual atmosphere (95%O<sub>2</sub>-5%H<sub>2</sub>O/10%H<sub>2</sub>-90%H<sub>2</sub>O) at 800 °C. The alloy exhibits a very good oxidation resistance compared to single atmosphere tests. However, a supplied electrical current significantly changes the nature of the oxides that form during the test. A very good Area Specific Resistance (ASR) parameter was measured in dual atmosphere, much lower than the values obtained in single atmosphere tests.

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## Introduction

High temperature water vapour electrolysis (HTE) is one of the most efficient technologies for mass hydrogen production. It works as an inverse fuel cell, using air at the anode side and water vapour at the cathode side. To increase hydrogen production, cells are overlapped, to create a stack. Stack construction requires the presence of interconnects, which physically connect the anode of one individual cell to the cathode of the neighbouring one.

The most important criteria required for the interconnect are a good oxidation resistance in air and in H<sub>2</sub>/H<sub>2</sub>O at high

temperature and a good electrical conductivity with an area specific resistance (ASR) below 0.1 Ω cm<sup>2</sup> for 25000 h (expected service lifetime) [1,2]. The targeted operating temperature of 800 °C enables the use of metallic interconnects and ferritic stainless steels seem to be the best option [3,23].

A few works have only been done in HTE conditions. As HTE is a reverse process compared to SOFC, literature data dealing with SOFC interconnects can be considered. Several works have been performed in O<sub>2</sub>/H<sub>2</sub>O gas environment, but only a few studies have been done under H<sub>2</sub>/H<sub>2</sub>O atmospheres. However, tests in dual atmosphere, by exposing the interconnect at the anode atmosphere at one side and at the

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<http://dx.doi.org/10.1016/j.ijhydene.2015.01.116>

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cathode atmosphere at the other side, are required in order to do a more realistic evaluation of the interconnect material. The SOFC literature reports very few studies done in dual atmosphere and they all suggest that the nature of the oxide scale that grows at the O<sub>2</sub> side is influenced by the H<sub>2</sub> gas present at the other side, while any evolution was not observed at the H<sub>2</sub> side [4–8].

In a previous study performed in HTE single atmospheres [9], the high temperature corrosion resistance and the electrical conductivity of a commercial ferritic stainless steel with 18% Cr-content, the K41X (AISI 441), were assessed in both anode (95%O<sub>2</sub>–5%H<sub>2</sub>O) and cathode atmosphere (10%H<sub>2</sub>–90% H<sub>2</sub>O) at 800 °C on as-rolled samples. In both atmospheres, the formation of a duplex oxide scale was revealed. In O<sub>2</sub>-H<sub>2</sub>O, a Cr-Mn spinel-type oxide grew at the top of an inner chromia layer, of about 0.2–1.4 μm after 100 h of exposition. In H<sub>2</sub>-H<sub>2</sub>O, an inner layer consisting of a Cr-Fe mixed oxide and an outer layer formed by a magnetite-type iron oxide were observed, with a total thickness of about 2–3 μm. The result obtained in single H<sub>2</sub>-H<sub>2</sub>O atmosphere was very surprising and not in agreement with the literature data concerning chromia-forming alloys in general [10] and K41X alloy in particular [11]. This specific behaviour could be related to the mechanical surface state [12].

In parallel, electrical resistivity measurements were performed in both HTE atmospheres during 100 h at 800 °C. A rather high, non-suitable, ASR parameter value (0.45 Ω cm<sup>2</sup>) was obtained in the cathode atmosphere, while a better behaviour, but still too high (0.1 Ω cm<sup>2</sup>) was observed in the anode atmosphere [13].

In this paper, we performed very new and innovative dual atmosphere tests in HTE conditions on the K41X material, the interconnect being exposed at one side to H<sub>2</sub>-H<sub>2</sub>O and at the other side to O<sub>2</sub>-H<sub>2</sub>O. In a first type of tests, the interconnect reactivity at 800 °C was evaluated in dual atmosphere and compared to single atmosphere results. In parallel, to better approach functioning situation, a second type of tests was done by applying an electrical current through the interconnect during the dual atmosphere exposition. The possible influence of the electrical current on the oxidation phenomena was checked by comparing microstructural analysis results obtained during the two different tests.

## Material and methods

The material selected for this study, K41X, is a commercial stainless steel with 18% Cr content (Fe–18%Cr–0.58%Si–0.52%Nb–0.25%Mn–0.14%Ti), provided by Aperam Isbergues, France. Prior to dual atmosphere tests, the as-rolled samples were cut into discs of 24 mm diameter and 1 mm thick. Specimens were ultrasonically cleaned in ethanol and dried at room temperature.

Dual atmosphere tests were performed at 800 °C by using a commercial set-up (ProboStat™), distributed by NorECs (Norwegian Electro Ceramics AS). The sample is placed between two Pt grid electrodes, that are assembled together by using a resort system. This assembly is placed on top of an alumina tube and then in the middle of a vertical furnace. The system's sealing is insured by a silver paste put between the sample

and the alumina tube. In this way, one surface of the sample is exposed at the anode atmosphere (95%O<sub>2</sub>–5%H<sub>2</sub>O) while the other surface is simultaneously exposed to the cathode atmosphere (10%H<sub>2</sub>–90%H<sub>2</sub>O). An electrical current can be applied and the resulting voltage can be measured. The Ohm's law allows to calculate the resistance of the growing oxide layer ( $R = V/I$ ). The ASR parameter is obtained by multiplying this resistance with the interconnect surface. These conductivity measurements can be performed in-situ during the dual atmosphere exposure of the interconnect. The value of the supplied electrical current was identical to single atmosphere tests, 150 mA, corresponding to a current density of 130 mA cm<sup>-2</sup>.

For safety reasons, on the cathode side of the set-up, H<sub>2</sub> was diluted in Ar and the real gas composition used was Ar-1% H<sub>2</sub>-9%H<sub>2</sub>O (that allows maintaining the H<sub>2</sub>-H<sub>2</sub>O ratio constant). Gas mixture (Ar/1%H<sub>2</sub> or O<sub>2</sub>) was passed through boiling distilled water. The H<sub>2</sub>O fraction (9% or 5% respectively) was controlled and adjusted by bubbling gas through a vertical tube cooled at a well-defined temperature.

The oxide surface and cross-section morphologies, as well as the chemical composition of the corrosion products, were analysed using a JEOL JSM-6400F scanning electron microscope. Phase composition of the oxide scales was determined by X-ray diffraction (XRD) with an Inel CPS 120 diffractometer using Cu Kα ( $\lambda = 0.154$  nm) radiation with a fixed incidence angle of 3°.

## Results and discussion

### Dual atmosphere influence on the oxidation behaviour (test without electrical current)

Fig. 1 shows the surface and cross-section morphologies of the K41X alloy after ageing during 100 h at 800 °C in dual atmosphere.

### Dual atmosphere influence at the O<sub>2</sub>-H<sub>2</sub>O side (HTE anode side)

At the anode side (Fig. 1a), the SEM images show a compact oxide layer, without any cracks or gap. The corrosion scale consists in some pyramidal grains randomly distributed on a continuous and compact layer made of smaller grains. The XRD pattern showed the presence of two oxides: (Cr,Mn)<sub>3</sub>O<sub>4</sub> and Cr<sub>2</sub>O<sub>3</sub>. Cross-section observations (Fig. 1b) together with EDX analysis confirm the presence of a duplex oxide scale: an inner scale formed by protective chromia and an outer scale mainly formed by the Cr-Mn spinel type oxide grains. The oxide scale is adherent to the substrate and its thickness is found to vary between 0.4 μm and 1.5 μm.

At the O<sub>2</sub>-H<sub>2</sub>O side of the dual atmosphere exposition, the oxide scale composition, morphology and thickness are very similar to those obtained in single atmosphere. This result is very interesting, as the few studies that were performed in dual SOFC conditions at 800 °C report an anomalous oxidation at the air side (dry air or moist air). The grown scales are significantly different of those grown on samples exposed to air on both sides [4,5]. This anomalous oxidation was related to the transport of hydrogen through the steel (that was proved to be over 60 mm in 1 h at 800 °C) [14]. In consequence,

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