Method to Measure Area Specific Resistance and Chromium Migration Simultaneously from Solid Oxide Fuel Cell Interconnect Materials

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Abstract

Chromium evaporation is identified as a major degradation mechanism in solid oxide fuel cell (SOFC) stacks. The major chromium source is the commonly used stainless steel interconnects, thus raising a need for protective coatings on the interconnect steel. Ex situ characterization methods of protective coatings involve chromium evaporation measurements, area specific resistance (ASR) measurements and long-term exposure tests. To replicate stack conditions, commonly used ASR measurement setups should be further developed. This work presents an improved characterization method for steels and coatings and aims to be an extension to state-of-the-art characterization methods. The studied steel samples, bare or coated, are placed adjacent to palladium foils with a screen-printed lanthanum-strontium-cobalt (LSC) layer and the resistivity over the pair is measured. The method offers similar contact materials, chromium migration mechanisms, electrical contacts and chemical interactions, as seen in stacks. Further, it enables post-test chromium migration analysis with electron microscopy. Demonstration of the method validated that protective coatings hindered both oxidation and chromium migration from the substrate steels. The presented method could aid in accelerating protective coating development.

Keywords: Chromium Poisoning, Ferritic Stainless Steel, Fuel Cells, Planar SOFC Stack, Protective Coating Development, Stack Degradation

1 Introduction

Chromia forming ferritic stainless steels have become the most common materials for interconnect plates in planar solid oxide fuel cell (SOFC) stacks [1, 2]. Examples of such steels are Crofer 22 H, Crofer 22 APU (ThyssenKrupp VDM) and ZMG 232 (Hitachi). At high temperatures, these steels form a double-layer at the surface to slow down the oxidation. The double-layer consists of chromium oxide and chromium-manganese oxide on top. These steels are suitable for mass manufacturing methods but require protective coatings to minimize corrosion and chromium evaporation in SOFC applications [1–4].

Chromium poisoning of the cathode is one of the major degradation mechanisms of SOFCs. In high temperatures and oxidizing atmospheres, volatile chromium species, such as CrO3 and CrO2(OH)2 compounds are formed [3]. The presence of steam enhances chromium volatility. The gaseous chromium species migrate to the triple phase boundary (TPB), where the chromium compounds form Cr2O3 or SrCrO4. At the triple phase boundary (TPB), chromia may block the cathode active sites, thus deterring the oxygen reduction reaction. [3, 5] Further, SrCrO4 is electrically insulating, which in combination with growing oxide layers on the interconnect steel surface leads to increased ASR, decreasing the stack performance. Chromium poisoning is a widespread degradation mechanism, as it has been observed from nickel-based, iron-based and chromium-based alloys [6].
Optimization of steel materials and protective coatings is thus a significant part in increasing the lifetime of SOFC stacks. Of the various coatings reported in literature, conductive spinels such as manganese-cobalt oxide and manganese-cobalt-iron oxide are the most promising alternatives due to their high conductivity, excellent chromium retention capability and a thermal expansion coefficient closely matching the one of the steel [1, 5]. Similar protective coatings are also studied for the use in solid oxide electrolyzer (SOE) stacks to prevent chromium migration [7–9].

The most realistic characterization method of steels and coatings is to perform long-term stack tests combined with post-test microscopy analysis [4, 10]. However, this requires extensive resources and is therefore optimal only for the validation phase. Therefore, various ex situ characterization methods are reported in literature to be used for screening and development purposes. These methods include for example Cr-vaporization measurements [11–16], long-term exposure tests, and area-specific resistance (ASR) measurements [17–20]. Such ex situ test setups should represent the conditions in SOFC stacks as closely as possible to have a good comparison between ex-situ and stack results. Especially in ASR-measurements, it is important to have realistic contact materials to obtain results representative for real stacks. For example, the contact materials’ mechanical properties, such as surface roughness, should be similar to the real situation.

State-of-the-art ex situ ASR measurements are typically performed using a symmetrical setup with two coated steel plates. Either these plates are in direct contact with each other or another material, such as platinum contact paste or lanthanum-strontium-manganese oxide (LSM) pellets, which differ from stack cathode layers in thickness and microstructure, is inserted between the steel plates. Such ex-situ test conditions differ from real stacks, in which the coated steel is in contact with the cathode, and the test results might not be fully representative for stack conditions. Therefore, the commonly used setups should be further developed to account for the above-mentioned factors and to give a more comprehensive picture of the coating performance.

This work contributes to the field by presenting a comprehensive characterization method for steels and coatings for SOFC applications. The method aims to be an extension of the state-of-the-art ASR measurements and enables evaluation of electrical resistance, chromium retention capability, and corrosion in conditions that simulate a real stack. The presented characterization method can be used with existing devices for state-of-the-art ASR measurements and the same sample is used for all above-mentioned measurements. In this setup, the coated steel samples are stacked adjacent to palladium foils with a screen-printed lanthanum-strontium-cobalt oxide (LSC) layer. Naturally, the LSC layer can be replaced with any other cathode material or one can use contact coatings to replicate the situation of interest. The setup offers the same contact materials, chromium migration mechanisms and electrical contacts, as in a real stack. It also enables simultaneous measurements of multiple samples. Only remaining disparities from stack conditions are the missing electrochemical reactions and dual-gas atmosphere, which to include require real stack tests. The method and its benefits are demonstrated in a characterization of coated and uncoated steel samples.

2 Experimental

2.1 Test Setup

The main components consist of coated steel plates and lanthanum-strontium-cobalt oxide (LSC) coated palladium plates, of which the coated steel plates are the components to be studied. The experimental setup is illustrated in Figure 1. The coated steel plates are stacked adjacent to the LSC coated palladium plates, which represent SOFC cathodes. The electrical resistance over each pair is measured with a four-wire technique. The setup simulates the stack conditions and offers the following advantages: (i) realistic electrical contact and chemical interactions between the protective coating and cathode material, and (ii) enabling post-test analysis of chromium migration through the coating by electron microscopy analysis of the adjacent LSC layer, as the coated steel is the main chromium source in the setup. Post-test microscopy analysis gives also information on coating microstructure, corrosion phenomena and chemical interaction with the cathode material.

The process of conventional characterization methods and the proposed method are compared graphically in Figure 2. In the presented method, the same sample is used for both ASR measurements and post-test scanning electron microscopy (SEM) to study corrosion phenomena and chromium migration into the cathode material. With commonly used characterization methods, ASR measurements are often performed separately. Conventional measurement setups might include symmetrical setups with two coated steel plates, possibly with Pt or Ag paste or LSM pellets in-between, or one plate coated on both sides with Pt paste [20–27]. Chromium evaporation is studied on separate samples with the so-called transpiration method where the volatile chromium is collected downstream of the samples with, for example, denuder tubes in which the volatile chromium reacts with Na2CO3 to form water-soluble Na2CrO4, from which the evaporated chromium amount can be analyzed [11, 12, 28]. These steel samples can later be analyzed by SEM. Our presented method is beneficial as it offers a comprehensive overview of the coating or steel performance within one setup.

![Fig. 1 Test setup. The steel plates coated on one side with Ce/Co were in contact with LSC coated palladium plates. Electrical resistance over the sample was measured. Note that thicknesses are not in scale but exaggerated for illustrational purposes.](image-url)
In this study, screen-printed LSC layers were 20 ± 2 μm thick and were produced by AS Elcogen on top of 0.5 mm thick palladium plates (Alfa Aesar, purity 99.9%). The LSC layers were screen-printed and sintered with identical procedures as used in the manufacturing of SOFC cells, so their electrical and mechanical properties are similar to actual SOFC cells manufactured by AS Elcogen. This ensures that electrical and mechanical contact between coated steel and LSC, as well as chromium poisoning and other cathode-side degradation phenomena replicate the situation in a real SOFC stack using AS Elcogen’s single cells. Palladium was chosen as the substrate material due to its low chromium content, its coefficient of thermal expansion (13.9 m/K), and its chemical stability at elevated temperatures. The Co coating, 10 nm Ce layer with 800 nm Co on top [30], aids to hinder chromia formation [31]. Studies by Froitzheim et al. [32] and Poitel et al. [33] show that the oxidation of the metallic Co layer is a fast process compared to the measurement times. Froitzheim have demonstrated that oxidation of a similar 640 nm metallic Co layer in 850 °C takes place in less than 30 s.

Dimensions of palladium and steel plates were 10 mm × 10 mm and they were equipped with 4 mm × 5 mm brackets with 0.5 mm holes for connection of voltage measurement wires. Measurement samples included three coated steel replicates and three uncoated steel replicates. Samples were wiped with ethanol before assembly. Steel and palladium plates were stacked between end plates (Crofer 22 APU), to which current wires were connected. As in stacks, the direction of the current is from the LSC layer to the steel. A compression force was applied by placing weights on the upper-most current collector. This setup was located inside a Nabertherm RS120 vertical tube furnace with a closed-end alumina working tube (Lilienthal, Germany). Bronkhorst EL-FLOW mass flow controllers and Controlled Evaporator Mixer (Ruurlo, The Netherlands) were used to control airflow rate and humidity inside the furnace. A Delta Elektronika ES 030-10 power source (Zierikzee, The Netherlands) was used to feed current through the samples. The current and voltage drops over samples were measured with Agilent 34970A data acquisition unit combined with Agilent 34901A Multiplexer (Santa Clara, United States) modules (uncertainties ± 5 μV, ± 0.5 mA).

### 2.2 Test Conditions

The test conditions were chosen to replicate typical operating conditions of planar anode supported SOFC stacks. The temperature was 700 °C. Heating and cooling rates were restricted to 10 °C h⁻¹ to ensure the durability of thin alumina tubes utilized as insulators for voltage leads. An airflow of 500 Nm³ min⁻¹ with added 15 Nm³ L⁻¹ min⁻¹ steam (3% humidity) was fed to the furnace to create a controlled atmosphere and simulate aggravated conditions, especially for chromium evaporation. Current density through the samples was 0.4 A cm⁻² and compression pressure was 0.4 MPa. Two tests with 100 and 2,000 h durations were conducted with separate samples to acquire information on corrosion phenomena and chromium retention over time.

### 2.3 Post-experimental Analysis

After ASR measurement, the samples were cast in epoxy resin and cross-sections were prepared for scanning electron microscopy. The goals of the post-experimental analysis were to measure the oxide layer thickness and the chromium content in the LSC layer. These data yield results on the ability of the coating to reduce oxidation of the steel and to prevent chromium transport from the steel to the cathode. The SEM used was a XL30-EBSP (FEI) equipped with an INCA Synergy 350 (Oxford instrument, Oxfordshire, United Kingdom) for energy-dispersive X-ray spectroscopy (EDS) measurement.

The oxide layer thickness was determined by measuring the area on the image corresponding to the oxide phase with the software ImageJ [35] and dividing it by the picture width according to the method described by Linder et al. [36].

| Table 1 | Chemical composition of Crofer 22 APU steel. Values in wt.%. [34]. |
|---------|---------------------------------------------------------------|
| Cr      | Fe    | C    | Mn   | Si   | Cu   | Al   | S    | P    | Ti   | La   |
| min.    | 20.0  | Bal. | 0.00 | 0.30 | 0.00 | 0.00 | 0.00 | 0.04 | 0.03 | 0.04 |
| max.    | 24.0  | 0.03 | 0.80 | 0.50 | 0.50 | 0.50 | 0.020| 0.050| 0.20 | 0.20 |
3 Results and Discussion

3.1 Area Specific Resistance

Resistances were measured between steel and palladium plates and they consist of the following contributions: resistance of bulk steel, resistance of bulk palladium, resistance of protective coating (including contact resistance to steel), contact resistance between protective coating and LSC, resistance of LSC (including contact resistance to palladium), and resistances of corrosion layers. Due to high electrical conductivities, bulk resistances of steel and palladium are negligible (−4 μΩ cm²) compared to others. Electrical conductivity of sintered LSC is approximately 85 cm⁻¹ at 700 °C [37], leading to a 0.25 m² cm⁻² area specific resistance of the LSC layer. Electrical conductivity of Co₃O₄ protective coating is 2.2 S cm⁻¹ at 700 °C [38], leading to 0.22 mΩ cm² ASR for a 5 μm thick layer. Measured resistance values are in the range of 5–45 mΩ cm², so most of the measured resistance consists of contact resistance between protective coating and LSC, and of resistance of corrosion layers. Recent results by Goebel et al. [39] also show that the contribution of the Co₃O₄ coating to ASR is negligible and the main part stems from chromium containing oxide layers.

A possible bias from thermoelectric effects was determined by measuring the voltage over the samples without feeding current through the samples. The measured voltages were less than 5 μV, which is in the same range as the uncertainty of voltage measurements. Thus, thermoelectric effects were found to be negligible.

Presented ASR values of the samples were calculated from measured current (I) and voltages (U) using Ohm’s law, and multiplied by sample areas (A) according to Equation (1)

\[ ASR = \frac{UA}{I} \]  

(1)

Uncertainty estimate for the ASR can be calculated as a total differential

\[ \Delta ASR = \frac{A}{I} \Delta U + \frac{U}{T} \Delta I + \frac{U}{I} \Delta A = ASR \left( \frac{\Delta U}{U} + \frac{\Delta I}{I} + \frac{\Delta A}{A} \right) \]  

(2)

Measured voltages over the samples were at minimum 2 mV (±5 μV), current was 0.4 A (±0.5 mA), and steel sample area was 1.0 cm². Samples were located manually with approximately 1 mm accuracy, leading to a mean sample area of 0.9 ± 0.1 cm². Uncertainty of ASR can therefore be estimated as

\[ \Delta ASR = ASR \left( \frac{5 \mu V}{2,000 \mu V} + \frac{0.5 mA}{400 mA} + \frac{0.1 cm²}{0.9 cm²} \right) \approx 0.11 \times ASR \]  

(3)

An additional uncertainty source is the thickness variation of LSC layer (20 ± 2 μm), but due to the high electrical conductivity of LSC, this variation has only a minor effect (0.02 mΩ cm²) on the ASR. Uncertainty estimate of ASR value is therefore approximately 11% of its value and its main source is the uncertainty in sample contact area.

Measured ASR values during the 2,000 h and 100 h test are illustrated in Figures 3 and 4. In both tests, three uncoated and three coated sample replicates were used but ASR values of one uncoated sample of the 100-hour test are omitted due to a measurement fault. In the end of the 2,000-hour test, the average ASR of coated samples is 12 mΩ cm² and the ASR of uncoated samples is 17 mΩ cm². Corresponding values in the end of the 100-hour test are 43 mΩ cm² for the uncoated samples and 53 mΩ cm² for the coated samples.

Figure 3 shows that the ASR values of uncoated samples are initially higher (45 mΩ cm²) and continuously decrease over the test period. The result is partially counter-intuitive, as one could expect the ASR to increase continuously with time due to the growth of oxide layers. However, this behavior was previously observed by Yang et al. [40] and Thomann et al. [4]. Yang et al. attributed the initial decrease of ASR to the growth of (Cr,Mn)₃O₄ spinel on top of the oxide layer, which improved the electrical contact. The relevance of this phenomenon to SOFC applications is limited because uncoated stainless steel interconnects are a source of chromium poisoning and are therefore unlikely to be utilized in SOFC stacks.
However, it would be beneficial to understand the ASR behavior to deepen the knowledge on ASR measurements.

3.2 Post-experimental Analysis

3.2.1 Microstructure and Oxide Layer Characterization

Post-experimental analysis was performed with scanning electron microscopy (SEM) in order to investigate: (i) the corrosion limiting properties of the protective coating by measuring the oxide layer thickness and its composition, (ii) the ability of the coating to reduce chromium migration from the steel to the screen-printed LSC layer, and (iii) the ASR behavior over time of the uncoated stainless steel.

Figure 5 presents four SEM cross-section micrographs of the interface between the steel sample and the screen-printed LSC layer. The gaps between the steel and the LSC layer are of different width in the different samples. These may stem from uneven sample surfaces or equally, from microscopy sample preparation, and no explicit conclusions may be drawn from these. After 100 h of testing, a reaction layer has already formed on top of the LSC layer in contact with the uncoated steel. The layer is composed of strontium chromate (SrCrO$_4$) and is 0.6 to 1.2 $\mu$m thick. Strontium chromate has been previously identified in the literature as an interaction product that plays a role in the chromium poisoning of LSCF cathode [3]. The degradation related to the formation of SrCrO$_4$ is not yet unambiguously understood. SrCrO$_4$ may increase the ASR because of its resistivity, which is relatively high (316.2 $\Omega$ m at 700 $^\circ$C [41]). Additionally, the SrCrO$_4$ may obstruct the path for reactant gas between the cathode surface and active layer. Lastly, the formation of SrCrO$_4$ causes a depletion of strontium in the cathode layer, which can result in an increase of the polarization resistance [42]. In the case of the coated steel samples, no SrCrO$_4$ was found at the interface between the coated steel and LSC surface, which is a sign that the coating significantly reduces chromium migration from the steel to the LSC layer. Further, formation of SrCrO$_4$ would not take place in state-of-the-art ASR measurement setups, in which for example, platinum paste is used and a real cathode counterpart is lacking. Thus, to observe such phenomena while evaluating protective coatings, the presented method should be helpful.

After 2,000 h of ASR testing, the oxide layers on both coated and uncoated steels have increased in thickness. The atomic composition of the oxide layer was investigated by EDS mapping, which is illustrated in Figure 6 for the case of a coated steel sample and Figure 7 for an uncoated steel sample. The oxide layer formed under the protective coating is 0.6 $\mu$m thick on average and is composed of chromium and manganese oxide, as seen in Figure 6. The internal oxidation nodules (marked with arrows in the manganese map, Figure 6d) are of the same composition. This result is in line with literature, as Crofer 22 APU is known to form a dual oxide layer of Cr$_3$O$_4$ and (Cr,Mn)$_3$O$_4$ [43]. A steel particle, where both iron and chromium are visible, has contaminated the LSC during...
sample preparation, as indicated by the arrow in the iron map (Figure 6c).

The oxidation layer on the uncoated steel is about 1.2 µm thick and is composed of chromium and manganese oxides (arrow in chromium map, Figure 7b). However, the oxide layer on the uncoated sample is very inhomogeneous in thickness, which suggests that a part of the oxide layer was removed during sample polishing and the actual thickness could be higher. Similar to the coated steel, the internal oxidation precipitates (shown by arrows in the manganese map, Figure 7d) are made of chromium and manganese oxides. Based on these results, the coating reduced the oxidation rate of the steel over the measurement time of 2,000 h.

3.2.2 Chromium Migration from Steel to LSC Layer

The ability of the coating to prevent chromium migration has been evaluated by measuring the chromium content of the adjacent LSC layer with EDS line scans. The results are presented in Figure 8. As shown in Figures 8a and 8b, EDS line scans are measured from the bulk steel below the oxide layer, over the oxide layer, over the coating if any, over the SrCrO₄ formation if any and over the complete LSC layer. The detection limit for chromium with the utilized equipment is about 1.0 atomic percent (at.%). Figure 8c and d show average Cr content in line scans of three coated and three uncoated steel sample replicates, after 100 h and 2,000 h of exposure. It is seen from Figure 8c that the chromium content of the LSC layers in contact with the coated steel is below the detection limit after 100 h of testing. The chromium content in the LSC layers adjacent to uncoated steel samples is 1–2 at.%. The difference in chromium content becomes very apparent after 2,000 h of testing, as illustrated in Figure 8d. The chromium content in the LSC layers is between 5 and 15 at.% for the uncoated steel samples and chromium is found through the whole LSC layers, whereas the values are still below 1 at.% for the coated steel samples. Some small amount of chromium, in the range of 1–2 at.%, is measured at the coating location; however, it is attributed to chromium signals from the steel itself because the interaction volume of the X-ray is of the same order of magnitude than the coating thickness (ca. 1 µm).

The transport of chromium from the steel to the LSC layer can also be confirmed by the chromium content in the substrate steel below the steel surface as illustrated in Figure 9. After 2,000 h at 700 ºC, the chromium content of coated steel is about 24 at.%, whereas this value is between 19 and 22 at.% for the uncoated steel. This result confirms the benefit of the coating with respect to chromium transport. The oxidation resistance of a stainless steel decreases with the chromium content and when the chromium content falls below about 16 wt.%, breakaway oxidation can occur [44]. This limit corresponds to about 17 at.% for the Crofer 22 APU steel used in this work.

3.2.3 ASR Behavior of the Uncoated Steel

ASR of the uncoated steel exhibits an initial increase until circa 100 h and then it continuously decreases over time, as seen in Figure 3. Yang et al. [40] observed the same phenomenon and attributed it to an improvement of the electrical contact due to the growth of (Cr,Mn)₃O₄ spinel on top of the oxide layer. Unfortunately, the quality of the electrical contact cannot be discussed from our observations because sample handling and preparation did not keep the physical contact of the different layers intact. However, the rapid growth of the strontium chromate layer (about 1 µm after 100 h, see Figure 5a) can explain a part of the rise of ASR in the first 100 h of the test. The resistivity of bulk strontium chromate has been measured to be 316.2 Ω m at 700 ºC [41]. Therefore, one µm of strontium chromate should cause an increase of ASR of about 2,900 mΩ cm². This value is significantly higher than our actual measured total ASR (about 45 mΩ cm²), which indicates that the strontium chromate layer is probably not uniform in thickness and there are paths free of strontium chromate. The decrease of the ASR between 200 to 2,000 h of test time can be explained by the growth of more conductive oxide such as chromium manganese spinel (Cr,Mn)₃O₄ (1.28 Ω m, [45]), as seen in Figure 7 and discussed in Section 3.2.1 and proposed by Yang et al. [40].

4 Conclusions

The presented characterization method offers a comprehensive method to evaluate the coating performance in terms of ASR, chromium retention properties and corrosion phenomena. The studied steel samples are placed adjacent to palladium counterparts with screen-printed real cathodes, which results in a setup with the same contact materials, chromium migration mechanisms and electrical con-
tacts as seen between the coated steel and cathodes in real stacks. In this work, a screen-printed LSC layer was used, but this can be replaced by another cathode material to simulate the situation of interest. The presented method is suited for characterization of steels, protective coatings as well as of the effect of different contact layers. This setup can be used with existing devices for conventional ASR measurements.

The characterization method was demonstrated with a 100- and 2,000-hour ASR measurement of both uncoated and coated Crofer 22 steel samples. The post-experimental SEM analysis of the ASR samples showed that the coating had a beneficial effect on the oxidation of the substrate steels and that the coating inhibited the chromium transport from the scale to the LSC layer. In addition, the setup enabled observation of other factors related to coating performance, such as formation of SrCrO₄ at the interface between uncoated steel and the LSC layer.

The aim for the development of the characterization method was to enable evaluation of electrical resistance, chromium retention capability, coating microstructure and corrosion of coated steels samples in conditions simulating a real stack with one single setup. Based on the results from the demonstration measurement, it is argued that the presented method achieved its purpose and, thus, enables to accelerate the development and characterization of metallic SOFC interconnects.

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**List of Symbols**

- **A** Area
- **ASR** Area specific resistance
- **BSE** Backscattered electrons
- **EDS** Energy-dispersive X-ray spectroscopy
- **I** Current
- **LSC** Lanthanum strontium cobalt oxide
- **LSM** Lanthanum strontium manganese oxide
- **SEM** Scanning electron microscope
- **SOE** Solid oxide electrolyser
- **SOFC** Solid oxide fuel cell
- **TPB** Triple phase boundary
- **U** Voltage

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