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Ni/Ce as a Cobalt-Free Coating Alternative for SOEC Interconnects

Effects of coating and substrate composition on oxidation, chromium evaporation, and dual-atmosphere corrosion

Master's thesis in Materials Science and Engineering .

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Abstract

Ferritic stainless steels are widely used as interconnect materials in solid oxide electrolysis cells (SOECs), but oxide-scale growth and chromium evaporation can reduce their durability. Although Co-based coatings effectively suppress chromium volatilisation, concerns regarding cobalt supply, cost, and sustainability motivate the development of cobalt-free alternatives.

This thesis evaluates Ni/Ce as a cobalt-free alternative to Co/Ce coatings on Crofer 22 APU and IM1 ferritic stainless steels. Coated and uncoated specimens were exposed in air containing 3% H₂O at 600 and 700 °C for up to 504 h. Oxidation behaviour, chromium evaporation, and oxide-scale development were investigated using mass-change measurements, the denuder technique, UV–Vis spectrophotometry, and SEM/EDX analysis. Dual-atmosphere exposures were also performed on IM1 at 600 °C.

Both coatings substantially reduced chromium evaporation compared with the corresponding uncoated materials. Co/Ce provided the strongest overall chromium-retention performance. The greatest improvement was observed for Co/Ce-coated Crofer 22 APU after 504 h at 700 °C, for which the cumulative chromium release was approximately 20.7 times lower than for the uncoated material. Ni/Ce also effectively suppressed chromium evaporation and showed promising performance as a cobalt-free coating. Ni/Ce-coated IM1 and Crofer 22 APU exhibited broadly similar oxidation behaviour, chromium-evaporation trends, and oxide-scale development, indicating that the coating had a strong influence on the degradation response under the investigated conditions.

Microstructural analysis revealed clear effects of coating chemistry and exposure environment on oxide-scale development. Under dual-atmosphere conditions, Co/Ce-coated IM1 exhibited the lowest local reaction-zone thickness and retained a compositionally distinct Co-rich outer region. In contrast, Ni/Ce-coated IM1 developed a thicker and more heterogeneous oxide/coating region, with Fe extending through much of the reaction zone.

Overall, Ni/Ce demonstrated potential as a cobalt-free coating for SOEC interconnects, but it did not provide the same overall level of protection as Co/Ce, particularly at 700 °C and under dual-atmosphere conditions. The results also indicate that lower-cost ferritic stainless steels such as IM1 may provide performance approaching that of conventional interconnect alloys when combined with a suitable protective coating.

Keywords: solid oxide electrolysis cell, ferritic stainless steel, interconnect, chromium evaporation, oxidation, Co/Ce coating, Ni/Ce coating, dual-atmosphere corrosion

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Tola Ahmed and Adam Abualdeber
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List of Acronyms

The acronyms used throughout this thesis are listed below in alphabetical order:

AEL	Alkaline electrolyser
BIB	Broad ion beam
BSE	Backscattered electrons
Co/Ce	Cobalt–cerium coating
EDX	Energy-dispersive X-ray spectroscopy
FSS	Ferritic stainless steel
LSM	Lanthanum strontium manganite
Ni/Ce	Nickel–cerium coating
PEM	Proton-exchange membrane
PVD	Physical vapour deposition
SE	Secondary electrons
SEM	Scanning electron microscopy
SOC	Solid oxide cell
SOEC	Solid oxide electrolysis cell
SOFC	Solid oxide fuel cell
TEC	Thermal expansion coefficient
TPB	Triple-phase boundary
UV–Vis	Ultraviolet–visible spectroscopy
YSZ	Yttria-stabilized zirconia

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1

Introduction

1.1 Background

Climate change is one of the major global challenges of the present century. Human activities, primarily through greenhouse-gas emissions, have caused global warming and increased the risk of severe environmental and societal impacts [1]. Since the energy sector remains a major contributor to global CO₂ emissions, the development of low-carbon energy technologies is essential for reducing the environmental impact of energy production and use [2].

Electricity generation from renewable sources such as wind and solar power has expanded rapidly in recent years. However, the intermittent nature of these energy sources creates challenges for electricity systems because electricity production does not always coincide with demand. Energy-storage technologies and flexible energy-system solutions are therefore needed to support grid stability and enable a greater share of renewable energy in the energy system [3].

Hydrogen is widely regarded as a promising energy carrier for addressing these challenges. It can store renewable electricity in chemical form and support the decarbonisation of sectors that are difficult to electrify directly [4]. However, most hydrogen is currently produced from fossil fuels, resulting in substantial CO₂ emissions [5]. Efficient and sustainable hydrogen-production technologies are therefore required to support the transition towards a low-carbon energy system.

Solid oxide cell (SOC) technology offers an efficient route for hydrogen production and energy conversion. When operated in solid oxide electrolysis cell (SOEC) mode, these systems produce hydrogen through high-temperature steam electrolysis. The elevated operating temperature improves reaction kinetics and allows part of the required energy to be supplied as heat rather than electricity, which can increase the overall process efficiency [6]. However, the high operating temperature and reactive gas environments also create materials challenges that can limit long-term durability [7].

Metallic interconnects are critical components in SOEC stacks because they provide electrical connections between adjacent cells while separating the fuel and oxidant gas compartments. Ferritic stainless steels (FSS) are commonly used as interconnect materials because of their suitable thermal expansion compatibility, good electrical conductivity, manufacturability, and relatively low cost [8, 9].

During operation, FSS interconnects form chromium-rich oxide scales. Although these scales can provide protection against continued oxidation, they may also release volatile chromium-containing species in humid oxidising environments [10]. These species can be transported through the gas channels and deposited on electrode surfaces, where they may contribute to performance degradation through chromium poisoning [11]. Continued oxide-scale growth can also increase the electrical resistance of the interconnect and reduce stack performance.

Protective coatings are therefore applied to metallic interconnects to reduce chromium evaporation, limit oxide-scale growth, and improve long-term stability [49]. Cobalt-based spinel coatings are widely used because they can effectively suppress chromium evaporation while maintaining suitable electrical conductivity [49, 13]. However, cobalt is considered a critical raw material and is associated with cost, supply, and sustainability concerns. These limitations have increased interest in cobalt-free coating alternatives for SOEC interconnect applications [13, 14].

In addition to coating composition, the underlying steel substrate can influence oxidation behaviour, chromium transport, oxide-scale formation, and corrosion resistance [15, 16]. Different FSS grades may therefore respond differently under otherwise similar exposure conditions, particularly in humid and dual-atmosphere environments relevant to SOEC operation [17].

Understanding the combined effects of substrate material, coating chemistry, coating thickness, temperature, and exposure environment is therefore important for improving interconnect durability. In the present work, Co/Ce and Ni/Ce physical vapour deposition (PVD) coatings are compared on Crofer 22 APU and IM1 to evaluate their effects on oxidation behaviour, chromium evaporation, and corrosion under SOEC-relevant conditions.

1.2 Problem Statement

FSS interconnects used in SOEC systems are exposed to high-temperature humid and dual-atmosphere environments that promote oxide-scale growth, chromium evaporation, and corrosion. These degradation processes can increase electrical resistance, contribute to chromium poisoning of the electrodes, and reduce stack durability.

Cobalt-based protective coatings are widely used because of their ability to suppress chromium volatilisation and improve interconnect stability. However, reliance on cobalt raises concerns related to material cost, supply risk, and long-term sustainability. In addition, specialised interconnect alloys such as Crofer 22 APU may

contribute to system cost, which creates interest in lower-cost FSS alternatives such as IM1.

Although coating chemistry, coating thickness, substrate material, temperature, and exposure environment are all known to influence interconnect degradation, their combined effects under SOEC-relevant humid-air and dual-atmosphere conditions are not yet fully understood. In particular, further investigation is required to determine whether cobalt-free coating systems can provide protection approaching that of conventional cobalt-based coatings and whether lower-cost FSS substrates can achieve comparable performance when combined with suitable protective coatings.

1.3 Research Objectives

This thesis investigates how substrate material, coating chemistry, coating thickness, temperature, and exposure environment influence the durability of FSS interconnects under SOEC-relevant humid-air and dual-atmosphere conditions. Particular emphasis is placed on oxidation behaviour, chromium evaporation, oxide-scale development, reaction-zone formation, and the potential use of lower-cost interconnect materials and cobalt-free coating systems.

The specific objectives are to:

- compare the oxidation behaviour and chromium-evaporation performance of Crofer 22 APU and IM1 under humid-air exposure at 600 and 700 °C;
- evaluate the ability of Co/Ce and Ni/Ce physical vapour deposition (PVD) coatings to modify oxidation behaviour and reduce chromium evaporation;
- assess the potential of Ni/Ce as a cobalt-free alternative to Co/Ce for SOEC interconnect applications;
- investigate the influence of substrate material on coating performance and determine whether the lower-cost IM1 alloy can approach the behaviour of the benchmark Crofer 22 APU alloy when protected by a suitable coating;
- examine the corrosion behaviour of coated and uncoated IM1 under dual-atmosphere exposure at 600 °C, with particular focus on surface morphology, oxide-scale development, reaction-zone formation, and degradation mechanisms;
- characterise exposed surfaces and broad ion beam (BIB)-prepared cross sections using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX);
- correlate the oxidation and chromium-evaporation results with the observed surface and cross-sectional microstructures.

2

Theory

2.1 Introduction to Solid Oxide Electrolysis Cells

Solid oxide electrolysis cells (SOECs) are high-temperature electrochemical devices that typically operate between 600 and 1000 °C [18]. Modern SOEC designs often target lower operating temperatures, typically around 600–900 °C, to improve durability and reduce material degradation. SOECs use electrical energy to produce chemical fuels by splitting steam into hydrogen (H₂) and oxygen (O₂). They can also be used to generate carbon monoxide (CO) or synthesis gas through CO₂ electrolysis or co-electrolysis [19].

An SOEC consists of two electrodes separated by an electrolyte that conducts ions while blocking electrons. When several cells are assembled into a stack, interconnects provide electrical connections between adjacent cells and separate the gas compartments. The system operates through coupled redox reactions driven by an external power supply. During operation, electrons flow through the external circuit, while ions migrate through the electrolyte [20].

Compared with conventional electrolysis technologies such as alkaline electrolysis (AEL) and proton-exchange membrane (PEM) electrolysis, SOECs operate at considerably higher temperatures. These elevated temperatures improve reaction kinetics and reduce the electrical energy demand for electrolysis.

The total energy required for steam electrolysis in an SOEC corresponds to the reaction enthalpy, ΔH , as expressed in Equation 2.1. In this relationship, ΔG represents the electrical-energy contribution, corresponding to the Gibbs free energy, while $T\Delta S$ represents the thermal-energy contribution associated with entropy.

$$\Delta H = \Delta G + T\Delta S \quad (2.1)$$

This temperature-dependent energy distribution is illustrated in Figure 2.1, which separates the total energy demand into electrical and thermal contributions. As the temperature increases, a larger fraction of the required energy can be supplied as heat, thereby reducing the electrical energy demand. This thermodynamic advantage enables high system efficiency, particularly when SOECs are integrated with high-temperature heat sources such as industrial waste heat or solar thermal energy. SOECs are therefore considered a promising technology for efficient and sustainable hydrogen production using renewable energy.

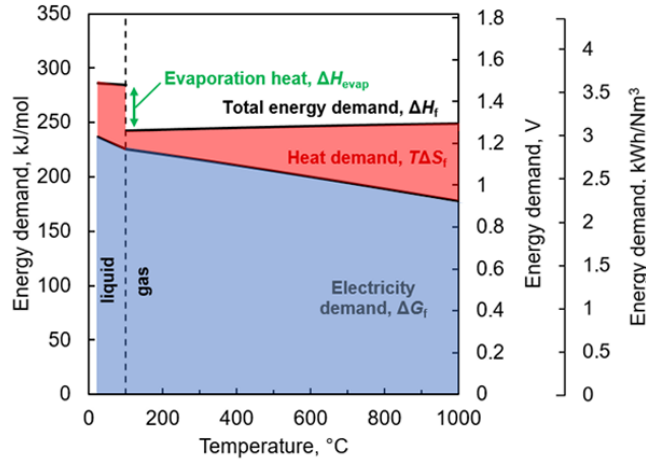


Figure 2.1: Energy splitting in a solid oxide electrolysis cell, illustrating the contributions of electrical and thermal energy [21].

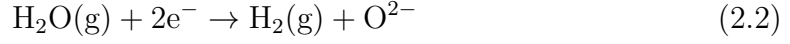
2.2 Operating Principle of Solid Oxide Electrolysis Cells

The operating principle of SOECs is based on high-temperature steam electrolysis, in which electrical and thermal energy are converted into chemical energy in the form of hydrogen [22]. SOECs use many of the same materials as solid oxide fuel cells (SOFCs), but operate in the reverse direction [23].

An SOEC consists of three main electrochemical components: the fuel electrode, the electrolyte, and the oxygen electrode. The fuel electrode, which acts as the cathode in electrolysis mode, is where steam is reduced to hydrogen gas and oxide ions, as shown in Equation 2.2. The electrolyte is a dense ion-conducting or proton-conducting ceramic positioned between the electrodes. In oxygen-ion-conducting SOECs, the electrolyte transports oxide ions, O^{2-} , from the fuel electrode to the oxygen electrode while preventing electronic conduction and gas mixing.

The oxygen electrode, which acts as the anode in electrolysis mode, oxidises the transported oxide ions to molecular oxygen while releasing electrons to the external circuit, as shown in Equation 2.3. The overall cell reaction is shown in Equation 2.4. The operating principle is illustrated in Figure 2.2.

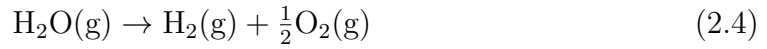
The electrochemical reaction at the fuel electrode is:



The electrochemical reaction at the oxygen electrode is:



The overall cell reaction is:



Electrons released at the oxygen electrode are transported through the metallic interconnects, which electrically connect the individual cells in the SOEC stack. At elevated operating temperatures, electrochemical reactions are accelerated and ionic conductivity increases. This reduces the electrical energy required for electrolysis and improves system efficiency. However, an external voltage is still required to drive the reaction [25]. These characteristics make SOECs attractive for applications in which high-temperature heat is available, such as nuclear reactors, concentrated solar-power systems, or industrial waste-heat sources.

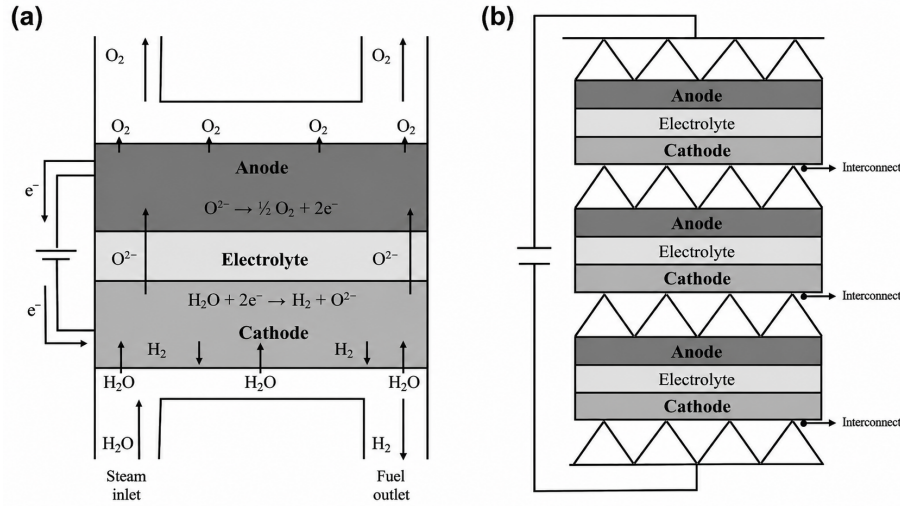


Figure 2.2: Schematic illustration of a solid oxide electrolysis cell: (a) single-cell operation showing steam reduction at the fuel electrode, oxide-ion transport through the electrolyte, and oxygen evolution at the oxygen electrode; and (b) a stacked-cell configuration with interconnects linking repeated fuel-electrode-electrolyte-oxygen-electrode units.

2.3 Solid Oxide Electrolysis Cell Materials and Designs

2.3.1 Electrolyte Materials

The electrolyte is a key component of an SOEC and can act as either an oxygen-ion conductor or a proton conductor. It must be chemically stable and have high ionic conductivity with minimal electronic leakage to ensure efficient operation. Electronic conduction reduces efficiency by interfering with ionic transport. The electrolyte should also be sufficiently thin to reduce ohmic losses and must prevent recombination of H_2 and O_2 [26].

Materials used in SOECs are largely derived from those used in SOFCs, an established commercial technology. The most common oxygen-ion-conducting electrolyte is yttria-stabilised zirconia (YSZ), typically used at temperatures above 700 °C. YSZ consists of ZrO_2 doped with approximately 8 mol% Y_2O_3 , providing high ionic conductivity, good mechanical strength, chemical stability, and relatively low cost. Other oxygen-ion-conducting electrolytes include doped LaGaO_3 and ceria-based oxides.

Proton-conducting ceramic electrolytes can also be used in SOECs. Kreuer [27] reviewed this approach in detail. Doped BaCeO_3 is considered a promising proton-conducting electrolyte, although its conductivity depends on the dopant type, dopant concentration, operating temperature, and oxygen partial pressure.

2.3.2 Electrode Materials

Electrode materials in SOECs must remain stable under reducing and oxidising environments while maintaining suitable microstructure, porosity, and thermal compatibility with the electrolyte. Adequate porosity is required for gas transport, while a sufficient density of triple-phase boundary (TPB) regions is needed to support the electrochemical reactions [28]. Matching the thermal expansion coefficients of the electrodes and electrolyte is also essential to minimise thermal stresses and maintain structural stability [29].

In SOEC operation, the fuel electrode acts as the cathode, where steam is reduced to produce hydrogen and oxide ions, as shown in Equation 2.2. Because the oxygen partial pressure on the fuel-electrode side is extremely low, approximately 10^{-12} to 10^{-16} bar, metallic materials such as Ni, Co, or Pt can be used [30].

Nickel is commonly used because of its high catalytic activity, good electrical conductivity, and relatively low cost. Since Ni is primarily an electronic conductor, it is typically combined with YSZ to form a porous Ni-YSZ cermet. This composite provides both electronic and ionic conduction while increasing the electrochemically active area. However, Ni-YSZ fuel electrodes may experience increased overpotentials during SOEC operation owing to Ni oxidation [31].

The oxygen electrode operates under highly oxidising conditions and is responsible for oxygen evolution. Oxygen-electrode materials therefore require high electronic conductivity, catalytic activity, and long-term thermal stability. Noble metals such as Pt and Au can be used; however, their high cost makes them impractical for large-scale SOEC applications [32]. Perovskite oxides, such as lanthanum strontium manganite (LSM), are widely used because of their good electrochemical performance [20].

The addition of YSZ to LSM improves TPB density and ionic transport [33]. However, extended high-temperature operation may promote interfacial diffusion of MnO_x from LSM into YSZ, leading to the formation of electrically insulating phases such as $\text{La}_2\text{Zr}_2\text{O}_7$ and SrZrO_3 . These phases can compromise long-term electrochemical stability [34]. This issue may be mitigated by slightly over-doping LSM with MnO_x , which can suppress the formation of secondary phases [35].

2.3.3 Interconnects

The main function of an interconnect in an SOEC stack is to provide an electrical pathway between adjacent cells. Specifically, the interconnect connects the oxygen electrode of one cell to the fuel electrode of the next cell while separating the fuel and oxygen gas compartments. When oxide ions migrate through the electrolyte and are oxidised at the oxygen electrode, electrons are released to the external circuit. These electrons pass through the interconnect and are transported to the fuel electrode of the adjacent cell.

Interconnect materials must therefore combine several demanding properties. They require high electronic conductivity, low ionic conductivity, good thermal conductivity, and a thermal expansion coefficient (TEC) compatible with the electrodes and electrolyte. They must also act as gas-tight separators to prevent mixing between the fuel and oxygen compartments [36].

Early interconnect materials were mainly ceramic. However, their high cost, brittleness, and fabrication difficulties made them less favourable for SOEC stack applications [37]. FSS interconnects have therefore received considerable attention because of their lower cost, ease of fabrication, good electrical and thermal conductivity, and TEC compatibility with other SOEC components.

Despite these advantages, FSS interconnects undergo corrosion during long-term operation at 600–1000 °C, particularly in the aggressive gas environments present on both sides of the interconnect. At high temperatures, these alloys form a dense chromium-rich oxide scale, mainly Cr_2O_3 , which provides some protection against further oxidation. However, chromium-containing species can volatilise from this scale and deposit on the electrodes, causing chromium poisoning. In addition, continued oxide-scale growth on unprotected alloy surfaces increases electrical resistance and reduces overall cell performance [38].

2.4 Oxidation of Metals

Oxidation is one of the main degradation processes affecting metallic materials at elevated temperatures. When a metal is exposed to an oxidising atmosphere, it can react with oxygen to form an oxide scale on the surface. The properties of this oxide scale strongly influence the subsequent oxidation behaviour of the material. Dense and adherent oxide scales can act as protective barriers by slowing the transport of reacting species, whereas porous, cracked, or poorly adherent scales provide limited protection.

The general oxidation reaction of a metal can be written as:



where M represents the metal and M_aO_b represents the oxide formed during oxidation [39].

In SOEC systems, metallic interconnects are exposed to high temperatures and reactive gas environments. Oxidation of these components can lead to oxide-scale growth, increased electrical resistance, chromium depletion, and reduced long-term performance. Understanding metal oxidation is therefore essential for interpreting the behaviour of coated and uncoated FSS interconnects investigated in this study.

2.4.1 Thermodynamics

The tendency of a metal to form an oxide is determined by the Gibbs free-energy change for the oxidation reaction. Under standard conditions, this is expressed as:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (2.6)$$

where ΔH° is the standard enthalpy change, ΔS° is the standard entropy change, and T is the absolute temperature. For metal oxidation, ΔH° is usually negative because oxide formation is exothermic. The entropy change is also often negative because gaseous oxygen is consumed during oxide formation. As a result, ΔG° generally becomes less negative as temperature increases, meaning that oxide stability decreases at higher temperatures [39, 40].

Oxidation is thermodynamically favourable when $\Delta G < 0$. When $\Delta G = 0$, the metal, oxide, and surrounding gas atmosphere are in equilibrium. Under non-standard conditions, the Gibbs free energy depends on the activities of the reacting species and the oxygen partial pressure:

$$\Delta G = \Delta G^\circ + RT \ln \left(\frac{a_{M_aO_b}}{a_M^a p_{O_2}^{b/2}} \right) \quad (2.7)$$

where a represents activity, R is the gas constant, and p_{O_2} is the oxygen partial pressure. For pure solid metals and oxides, the activities of the solid phases can be approximated as unity. The equation can therefore be simplified to:

$$\Delta G = \Delta G^\circ - \frac{b}{2} RT \ln p_{O_2} \quad (2.8)$$

This relationship shows that oxidation depends on both temperature and oxygen partial pressure. A higher oxygen partial pressure increases the thermodynamic driving force for oxide formation, while increasing temperature generally reduces oxide stability for many metal oxides.

The relationship between oxide stability, temperature, and oxygen partial pressure is commonly represented using Ellingham diagrams. These diagrams plot the standard Gibbs free energy of oxide formation as a function of temperature and are useful for comparing the relative stability of different oxides [40, 41].

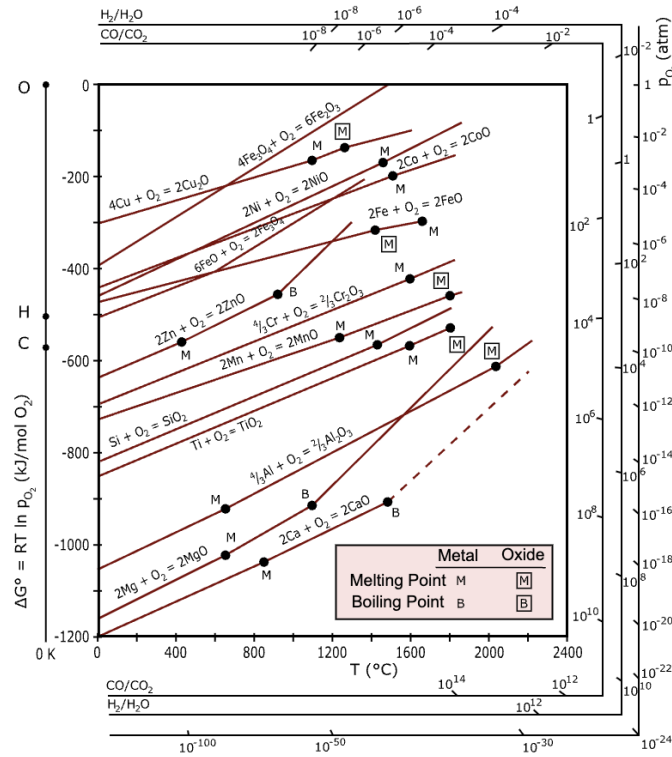


Figure 2.3: Ellingham diagram showing the temperature dependence of ΔG° for oxide formation [42].

2.4.2 Oxide-Growth Mechanism

The initial stages of metal oxidation involve several steps, as illustrated in Figure 2.4. When a metal is exposed to an oxidising atmosphere at elevated temperature, oxygen molecules first adsorb on the metal surface. The adsorbed oxygen then reacts with metal atoms to form oxide nuclei. As oxidation continues, these nuclei grow

laterally and vertically until they coalesce into a continuous oxide scale covering the surface [39, 40].

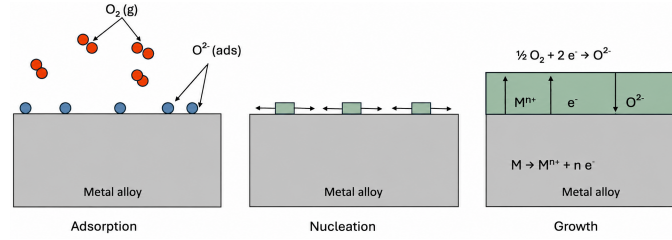


Figure 2.4: Schematic illustration of oxide formation on a metal surface, showing oxygen adsorption, oxide nucleation, and growth of a continuous oxide layer.

Once a compact and continuous oxide scale has formed, further growth is often controlled by the transport of reacting species through the oxide. According to classical oxidation theory, often referred to as Wagner theory, the growth of a compact and adherent oxide scale requires the coupled transport of ions and electronic charge carriers through the scale [39, 40]. Depending on the oxide composition and defect structure, metal ions, oxygen ions, or both may migrate through the oxide. As a result, oxide growth can occur at the metal–oxide interface, at the oxide–gas interface, or at both interfaces.

The transport properties of oxide scales are strongly influenced by their defect structure. Many oxides are non-stoichiometric and contain point defects, such as vacancies and interstitials, which provide pathways for ionic transport. At the same time, electronic defects, such as electrons or electron holes, must move through the oxide to maintain charge neutrality and allow the oxidation reaction to continue as the scale thickens [40].

For FSS interconnects, oxidation typically results in the formation of chromium-rich oxide scales. These chromia scales can provide protection by limiting additional oxidation. However, continued scale growth increases the electrical resistance of the interconnect and may contribute to long-term performance losses [8, 10]. The oxide-scale structure also influences chromium transport towards the surface, which is closely related to chromium evaporation. Understanding oxide-growth mechanisms is therefore important for interpreting the oxidation behaviour of coated and uncoated interconnect materials in this study.

2.4.3 Oxidation Kinetics

Thermodynamics determines whether oxide formation is favourable, whereas oxidation kinetics describes the rate at which an oxide scale grows. For SOEC interconnects operating at elevated temperatures, oxidation kinetics is important because oxide-scale growth can increase electrical resistance and contribute to long-term degradation.

After an initial oxide layer has formed, the oxidation rate depends on the protectiveness of the scale and on the rate-controlling transport processes. Depending on the

oxide properties and exposure conditions, oxidation may follow linear, parabolic, or logarithmic growth behaviour, as illustrated in Figure 2.5 [39, 40].

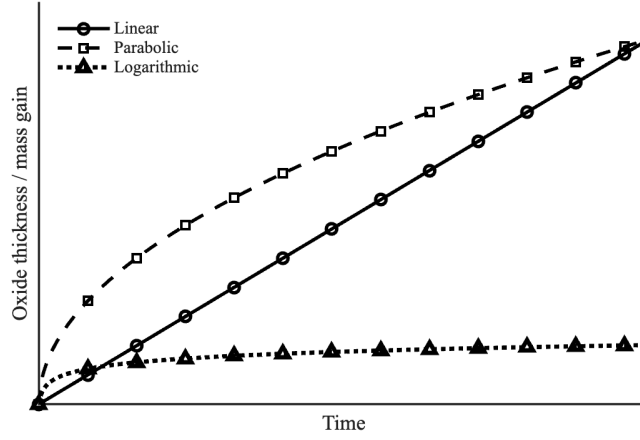


Figure 2.5: Schematic comparison of linear, parabolic, and logarithmic oxidation kinetics.

Linear oxidation is commonly associated with non-protective oxide scales that are porous, cracked, or poorly adherent. In this case, the oxide does not act as an effective diffusion barrier, allowing oxidising species to reach the metal surface continuously. The oxidation rate therefore remains approximately constant and can be described by:

$$x = k_l t + C \quad (2.9)$$

where x is the oxide thickness, k_l is the linear rate constant, t is the exposure time, and C is a constant related to the initial oxide thickness [39, 40].

When a dense and protective oxide scale forms, oxidation is often controlled by diffusion through the growing oxide layer. In this case, the oxidation rate decreases with time because the diffusion distance increases as the scale becomes thicker. This behaviour is described by the parabolic rate law:

$$x^2 = k_p t + C \quad (2.10)$$

where k_p is the parabolic rate constant. Parabolic oxidation behaviour is commonly observed for protective oxide scales, including chromia scales formed on FSS [39, 40].

At lower temperatures or during the early stages of oxidation, when the oxide film is very thin, oxidation may follow logarithmic behaviour:

$$x = k_{\log} \log(t + t_0) + C \quad (2.11)$$

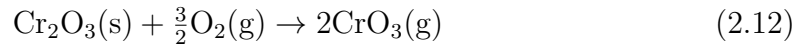
where $k_{\log}$, t_0 , and C are constants. Logarithmic oxidation is usually associated with thin oxide films, in which short diffusion distances or electric-field effects can influence transport through the scale [39, 40].

In practice, oxidation behaviour may deviate from ideal rate laws because several processes can occur simultaneously. For FSS interconnects, chromia-scale growth may be accompanied by chromium evaporation, coating–substrate interactions, changes in oxide-scale structure, and chromium depletion near the surface. These effects are important when interpreting the oxidation behaviour of coated and uncoated SOEC interconnect materials [10].

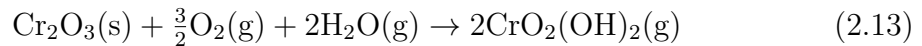
2.4.4 Chromium Evaporation

Chromium evaporation is a major degradation mechanism in FSS interconnects used in solid oxide electrolysis and fuel-cell systems at high temperatures. These steels form a chromium-rich oxide scale, mainly Cr_2O_3 , which slows oxidation. However, in atmospheres containing oxygen and water vapour, this scale can react to form volatile chromium species, leading to chromium loss and degradation of the protective oxide layer [10, 43].

At elevated temperatures, chromium oxide can react with oxygen to form gaseous chromium trioxide:



In humid environments, chromium oxyhydroxide species can form. Under SOEC-relevant humid oxidising conditions, a dominant volatile species is chromium oxyhydroxide:



Water vapour stabilises these species and increases chromium volatility [43, 10]. The gaseous chromium species can be transported through interconnect channels and deposited on electrode surfaces, particularly the oxygen electrodes. These deposits can block active reaction sites and reduce cell performance, a phenomenon known as chromium poisoning [11].

Protective coatings are therefore applied to interconnect materials to reduce chromium evaporation while maintaining electrical conductivity and improving long-term durability in SOEC environments [10, 49].

2.4.5 Protective Coatings

Protective coatings are applied to FSS interconnects to reduce chromium evaporation, limit oxide-scale growth, and maintain low electrical resistance during SOEC operation. At high temperatures and in humid oxidising atmospheres, volatile chromium species can form from the chromia scale and deposit on cell components, contributing to performance degradation. Coatings therefore play an important role in reducing chromium release and improving interconnect stability [49].

These coatings can act as diffusion barriers by slowing chromium transport from the steel substrate to the gas phase. In addition, they may react with chromium to form stable oxide or spinel phases, thereby reducing the formation of volatile chromium species [49].

Comparative studies have shown that Mn–Co-based spinel coatings provide highly effective chromium suppression. Chromium evaporation can be reduced by more than an order of magnitude, while oxide scales remain thin and area-specific resistance remains low during long-term exposure [49]. For this reason, Mn–Co spinel coatings are commonly used as benchmark materials for interconnect coatings.

However, cobalt is considered a critical raw material, which has increased interest in cobalt-free alternatives [13, 14]. Nickel-based coatings are one potential option. Ce/FeNi coatings have shown lower oxidation rates and improved electrical performance compared with Ce/Co coatings, although chromium evaporation may remain higher than for Co-containing coatings [14].

Cerium additions can further improve coating performance by influencing oxide-scale growth and adhesion. Ce-containing coatings have been reported to reduce chromium evaporation when good surface coverage is achieved [44].

During SOEC operation, protective coatings must remain stable at high temperature, in humid atmospheres, and under dual-atmosphere conditions. Coating integrity and phase stability are therefore important for long-term interconnect performance [49, 14]. These challenges motivate the comparison of Co/Ce and Ni/Ce coatings in the present work.

2.5 Dual-Atmosphere Conditions

In SOCs, metallic interconnects operate under dual-atmosphere conditions, with an oxygen-containing gas on the air side and a hydrogen–steam mixture on the fuel side. This creates a large oxygen partial-pressure gradient across the material, which can influence oxidation behaviour. FSS interconnects typically form a chromium-rich oxide scale, mainly Cr_2O_3 , which can slow further oxidation. However, corrosion may become more severe when different atmospheres are present on opposite sides of the material, a phenomenon known as the dual-atmosphere effect [17].

One proposed explanation for this effect is that hydrogen from the fuel side can diffuse through the steel and influence oxide-scale formation on the air side, leading to accelerated corrosion and reduced scale protectiveness. These processes can

reduce interconnect durability and contribute to chromium-related degradation in SOEC systems [45, 38]. Dual-atmosphere exposure tests are therefore used to evaluate the corrosion behaviour and long-term stability of interconnect materials under conditions that better represent SOC operation.

3

Materials and Methods

This chapter describes the investigated ferritic stainless-steel grades, the protective coating systems, the SOEC-relevant exposure conditions, and the characterisation methods used after exposure.

3.1 Materials

Two FSS grades, Crofer 22 APU and IM1, were investigated in this study. Both materials were supplied as sheet material and cut into different specimen geometries depending on the exposure type. The chemical compositions of the materials are presented in Table 3.1.

Table 3.1: Chemical compositions of IM1 and Crofer 22 APU (wt.%).

Material	Fe	Cr	C	Mn	Si	Ni	Ti	Nb	Al	Cu	Mo	V	N	P	S
IM1	Bal.	20.96	0.029	–	0.09	–	–	–	0.022	–	–	–	–	–	0.0006
Crofer 22 APU	Bal.	22.8	0.002	0.45	0.01	–	0.07	–	0.01	0.01	–	–	–	0.002	<0.002

Bal. = balance; – = not specified or below the reporting limit.

Square coupons with dimensions of 15×15 mm were used for the single-atmosphere oxidation and chromium-evaporation experiments, while disc-shaped specimens with a diameter of 21 mm were used for the dual-atmosphere exposures. Before exposure, the specimens were ultrasonically cleaned in acetone for 20 min, followed by ethanol for 20 min, and then dried in air. Both coated and uncoated specimens were included in the study.

3.2 Coatings

Two PVD coating systems, Co/Ce and Ni/Ce, were investigated. The coated specimens were supplied with pre-deposited bilayer coatings on the FSS substrates. Both coating systems consisted of a Ce layer adjacent to the substrate, followed by either a Co or Ni top layer. The coating sequences are therefore referred to as substrate/Ce/Co and substrate/Ce/Ni, respectively.

The layer thicknesses were verified using cross-sectional SEM. The Ce layer was 10 nm thick. For the Co/Ce coating, the Co top layer was 600 nm thick on Crofer 22 APU and 300 nm thick on IM1. For the Ni/Ce coating, the Ni top layer was 1000 nm thick on both substrates.

Because the coated specimens were supplied with pre-deposited coatings, detailed deposition parameters were not available. The coated and uncoated specimens were prepared according to the exposure conditions summarised in Table 3.2.

3.3 Exposures

Table 3.2 summarises the exposure conditions used in this study. The tests were designed to represent SOEC-relevant environments, including humid air and dual-atmosphere conditions with a hydrogen-containing fuel-side atmosphere.

Both coated and uncoated Crofer 22 APU and IM1 specimens were tested to evaluate the effects of substrate material, coating chemistry, coating thickness, temperature, and exposure environment on oxidation behaviour, chromium evaporation, and corrosion performance.

Table 3.2: Exposure conditions used for Crofer 22 APU and IM1 specimens.

Exposure	Atmosphere	Temp. (°C)	Surface configuration	Dimensions
Air-side oxidation	Air + 3% H ₂ O	600, 700	Uncoated	15 × 15 mm
		600, 700	Co/Ce // Co/Ce	
		600, 700	Ni/Ce // Ni/Ce	
Air-side chromium evaporation	Air + 3% H ₂ O	600, 700	Uncoated	15 × 15 mm
		600, 700	Co/Ce // Co/Ce	
		600, 700	Ni/Ce // Ni/Ce	
Dual-atmosphere exposure	Air + 3% H ₂ O // Ar + 5% H ₂ + 3% H ₂ O	600	Uncoated // Uncoated	∅ 21 mm
		600	Co/Ce // Co/Ce	
		600	Ni/Ce // Ni/Ce	

The single-atmosphere exposure durations were 24, 168, and 504 h. The dual-atmosphere specimens were examined after 168 h and after a total exposure time of 504 h. The notation // indicates the surface condition or atmosphere on opposite sides of the specimen.

3.3.1 Oxidation

Single-atmosphere oxidation experiments were conducted in air containing 3% H₂O at 600 and 700 °C using a tubular furnace. The airflow rate was set to 6000 standard cubic centimetres per minute (sccm). The exposure times were 24, 168, and 504 h.

The specimens were weighed before and after exposure using a METTLER TOLEDO XP6 microbalance to determine the mass change. The equipment used for the oxidation experiments is shown in Figure 3.1.



(a) Tubular furnace used for the oxidation experiments.



(b) METTLER TOLEDO XP6 microbalance used for mass measurements.

Figure 3.1: Experimental equipment used for the oxidation experiments.

For each exposure condition, two specimens were exposed, and the measured mass changes were averaged. Separate specimens were used for each exposure duration. The average mass change was normalised by the total exposed surface area to obtain the mass change per unit area, $\Delta m/A$, according to

$$\frac{\Delta m}{A} = \frac{m_t - m_0}{A}, \quad (3.1)$$

where m_0 is the initial mass, m_t is the mass after exposure, and A is the total exposed surface area. The resulting $\Delta m/A$ values were used to compare the oxidation behaviour of the coated and uncoated specimens.

3.3.2 Cr(VI) Evaporation

Chromium evaporation was measured using the denuder technique, which has been widely used to investigate chromium volatilisation from Fe–Cr interconnect materials [46]. The specimens were exposed in a tubular furnace under the same atmosphere and temperature conditions described in Section 3.3.1.

During exposure, volatile chromium species formed at the specimen surface were transported downstream by the gas flow. The denuder tube was replaced after each exposure interval, allowing the amount of chromium collected during each interval to be determined separately.

A denuder tube coated with sodium carbonate, Na_2CO_3 , was placed downstream of the specimens to collect the volatile chromium species described in Section 2.4.4. These species react with the sodium carbonate coating to form solid sodium chromate, Na_2CrO_4 , according to



After each exposure interval, the denuder tube and wash bottle were rinsed with deionised water to dissolve the collected sodium chromate. The chromium-containing solutions were then analysed using ultraviolet–visible (UV–Vis) spectrophotometry.

The chromate concentration was calculated from the measured absorbance using the calibration factor obtained from the calibration procedure:

$$c = \frac{\text{Abs}}{0.404}, \quad (3.3)$$

where c is the chromate concentration, Abs is the measured absorbance, and 0.404 is the calibration factor.

The total chromate concentration was obtained by adding the contributions from the denuder solution and the wash-bottle solution:

$$c_{\text{total}} = c_{\text{den}} + c_{\text{bot}}, \quad (3.4)$$

where c_{den} and c_{bot} are the chromate concentrations in the denuder and wash-bottle solutions, respectively.

The amount of chromate collected during each exposure interval was calculated as

$$m_{\text{CrO}_4^{2-}} = c_{\text{total}}V, \quad (3.5)$$

where $V = 0.025$ L is the solution volume.

The corresponding chromium amount was calculated using the molar-mass ratio between Cr and CrO_4^{2-} :

$$m_{\text{Cr}} = m_{\text{CrO}_4^{2-}} \frac{52}{116}. \quad (3.6)$$

The chromium release was normalised by the exposed surface area according to

$$\text{Cr}_{\text{release}} = \frac{m_{\text{Cr}}}{A}, \quad (3.7)$$

where A is the exposed surface area.

The cumulative chromium release was obtained by summing the chromium-release values from the individual exposure intervals:

$$Cr_{cum} = \sum Cr_{release}. \quad (3.8)$$

The interval chromium-evaporation rate was calculated by dividing the chromium release measured during each interval by the corresponding interval duration.

Figure 3.2 illustrates the experimental setup used for the chromium-evaporation measurements.

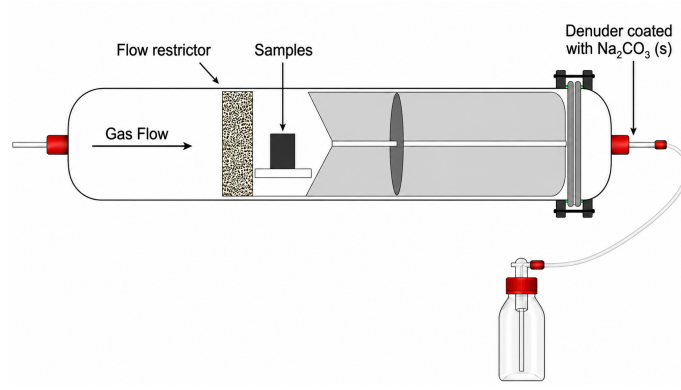


Figure 3.2: Schematic illustration of the chromium-evaporation measurement system, adapted from Froitzheim et al. [46].

3.3.3 Dual-Atmosphere Exposure

IM1 specimens were exposed under dual-atmosphere conditions at 600 °C. Air containing 3% H₂O was supplied to one side of each specimen, while Ar containing 5% H₂ and 3% H₂O was supplied to the opposite side. Before exposure, the specimens were cleaned according to the procedure described in Section 3.1.

The dual-atmosphere conditions were established using a specimen holder based on the designs described by Holcomb et al. and Gannon et al. [47, 48]. Gold rings were used to seal the specimens between the two gas environments. The specimen holder used for the exposure experiments is shown in Figure 3.3.

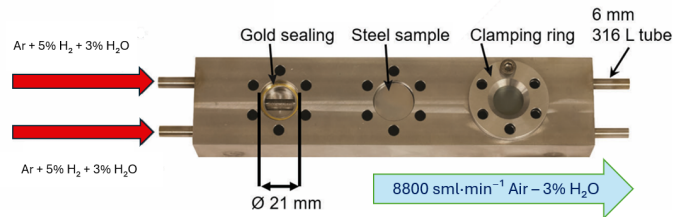


Figure 3.3: Specimen holder used for the dual-atmosphere exposure experiments [49].

The specimens were exposed for 168 h and then removed for photographic documentation. The exposure was subsequently continued for an additional 336 h, resulting in a total exposure time of 504 h.

After exposure, selected cross sections were prepared using BIB milling to obtain smooth surfaces with minimal preparation-induced damage. Both the exposed specimen surfaces and the BIB-prepared cross sections were examined using SEM to evaluate surface morphology, oxide-scale morphology, and coating/substrate features.

3.4 Characterisation Methods

The specimens exposed under the different test conditions were characterised to evaluate oxide-scale formation, coating stability, chromium evaporation, and microstructural changes. Surface and cross-sectional analyses were performed using SEM/EDX, while chromium collected using the denuder setup was quantified using UV–Vis spectrophotometry. Cross sections of selected specimens were prepared using BIB milling to obtain smooth surfaces suitable for detailed oxide-scale analysis.

3.4.1 Scanning Electron Microscopy and Energy-Dispersive X-ray Spectroscopy

SEM was used to examine the surface morphology and cross-sectional microstructure of the specimens after exposure. The technique produces high-resolution images by scanning the specimen surface with a focused electron beam. Interactions between the electron beam and the specimen generate several signals, including secondary electrons (SE), backscattered electrons (BSE), and characteristic X-rays. These signals provide information about surface morphology, compositional contrast, and elemental composition [50, 51]. The principle of SEM is illustrated in Figure 3.4.

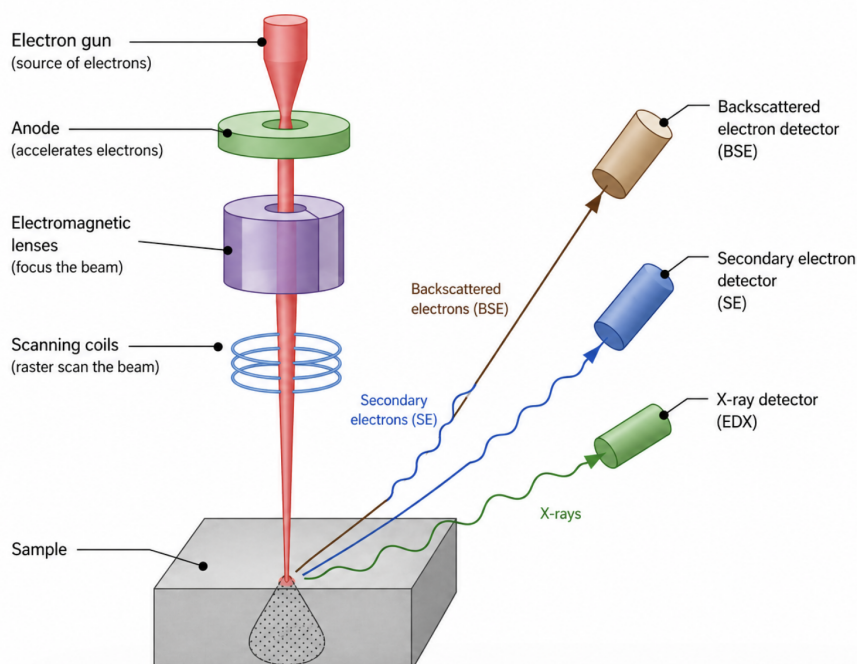


Figure 3.4: Schematic illustration of the SEM principle, showing the generation of secondary electrons, backscattered electrons, and characteristic X-rays during electron–specimen interaction.

Secondary electrons are low-energy electrons generated close to the specimen surface. They are mainly used to image surface features such as roughness, grain morphology, and oxide-scale formation. Because they originate from a shallow interaction depth, SE images provide detailed information about surface topography [50].

Backscattered electrons are primary electrons that are scattered out of the specimen following elastic interactions with atoms in the material. The BSE signal is sensitive to atomic number, meaning that regions containing elements with higher atomic numbers generally appear brighter than regions containing lighter elements. BSE imaging was therefore used to identify compositional contrast between oxide scales, coatings, and the steel substrate [52].

EDX was used to analyse the elemental composition of selected regions. Characteristic X-rays produced during electron–specimen interactions were used to identify the elements present in the surface and cross-sectional regions of interest [52].

3.4.2 Ultraviolet–Visible Spectrophotometry

UV–Vis spectrophotometry was used to determine chromium concentrations in aqueous solutions obtained after rinsing the denuder tube and wash bottle. The collected solutions may contain different hexavalent chromium species, Cr(VI), including chromate and dichromate ions.

Under alkaline conditions at $\text{pH} \geq 9$, chromate ions, CrO_4^{2-} , are the dominant Cr(VI) species, accounting for at least 99.6% of the dissolved Cr(VI) [53]. The pH

of all collected solutions was therefore measured. When the pH was below 9, small quantities of 0.1 M NaOH were added to ensure alkaline conditions and promote the predominance of chromate ions.

The chromate concentrations were determined using the Beer–Lambert law:

$$A = \log_{10} \left(\frac{I_0}{I} \right) = \varepsilon_{\lambda} c l, \quad (3.9)$$

where A is the absorbance, I_0 and I are the incident and transmitted light intensities, respectively, ε_{λ} is the molar absorption coefficient at wavelength λ , c is the concentration of the absorbing species, and l is the optical path length.

The spectrophotometric measurement setup used for the absorbance measurements is illustrated in Figure 3.5.

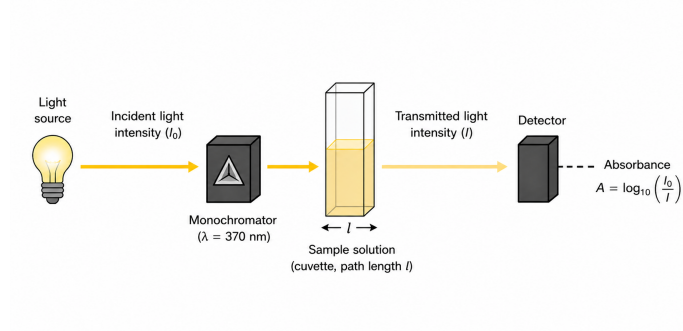


Figure 3.5: Schematic illustration of the spectrophotometric measurement setup used to determine chromate concentration at 370 nm.

Chromate ions exhibit strong absorbance at 370 nm in aqueous solution; therefore, all measurements were performed at this wavelength. The molar absorption coefficient was determined from a calibration curve prepared using standard chromate solutions of known concentration.

3.4.3 Broad Ion Beam Milling

Cross sections of selected specimens were prepared by BIB milling using a Leica EM TIC 3X instrument. Before ion-beam preparation, each specimen was sectioned, and a silicon wafer was attached to provide mechanical support. The specimen surface was then mechanically polished to obtain a clean and smooth starting surface.

During BIB milling, material is gradually removed by accelerated argon ions, thereby exposing a clean cross section. This technique produces smooth cross-sectional surfaces with minimal preparation-induced damage, making it suitable for microstructural analysis of oxide scales and coatings.

Compared with conventional mechanical polishing, BIB preparation reduces deformation and smearing, particularly in thin oxide layers and coatings. The prepared cross sections were subsequently examined using SEM/EDX.

4

Results and Discussion

4.1 Oxidation Behaviour and Chromium Evaporation

The oxidation behaviour and chromium evaporation of uncoated and coated IM1 and Crofer 22 APU were investigated after exposure in humid air for 24, 168, and 504 h at 600 and 700 °C. The investigated coating systems were Co/Ce and Ni/Ce bilayers. The metallic top-layer thicknesses were not identical: the Co layer was approximately 300 nm on IM1 and 600 nm on Crofer 22 APU, whereas the Ni layer was approximately 1000 nm on both substrates. Consequently, the observed differences between the coated specimens reflect the combined effects of coating chemistry, coating thickness, substrate material, and exposure temperature.

Separate experimental setups were used to evaluate oxide-scale growth and chromium volatilisation. Oxidation behaviour was assessed from the mass change normalised by the exposed surface area, whereas chromium evaporation was quantified from the accumulated chromium release measured during exposure. Together, these measurements provide insight into the high-temperature corrosion behaviour and protective performance of the investigated coating systems.

4.1.1 Net Mass Gain

As shown in Figure 4.1, the coated specimens generally showed higher net mass gain than the uncoated specimens at both temperatures. Among the coated conditions, Ni/Ce showed the highest mass gain, followed by Co/Ce. For example, at 700 °C, Ni/Ce-coated IM1 reached approximately 0.29 mg/cm² after 504 h, compared with approximately 0.18 mg/cm² for Co/Ce-coated IM1. A similar trend was observed for Crofer 22 APU.

The higher mass gain of the Ni/Ce-coated specimens is consistent with greater overall oxidation of the coating/substrate system. However, this difference cannot be attributed to coating chemistry alone. The Ni top layer was approximately 1000 nm thick on both substrates, whereas the Co top layer was approximately 300 nm on

IM1 and 600 nm on Crofer 22 APU. The larger amount of Ni-containing coating material available for oxidation may therefore have contributed to the higher measured mass gain. In contrast, the lower and more gradual mass increase of the Co/Ce-coated specimens is consistent with more limited overall oxidation under the present conditions, although coating thickness must also be considered when comparing the two systems.

The time dependence of the coated specimens further supports this interpretation. Ni/Ce-coated specimens showed a rapid increase in mass during the early stages of exposure, followed by a tendency to level off. Co/Ce-coated specimens, on the other hand, showed a more gradual increase in mass with exposure time. This behaviour is consistent with the progressive development of an oxide layer that increasingly limits the transport of reacting species, as discussed in Section 2.4.3.

In contrast, the uncoated specimens showed very low or, in some cases, negative net mass changes. For example, uncoated IM1 exposed at 600 °C decreased to approximately -0.035 mg/cm^2 after 504 h. This behaviour can be explained by the competing effects of oxide formation and chromium evaporation. While oxide formation contributes to mass gain, chromium volatilisation leads to mass loss. At longer exposure times, the mass loss associated with chromium evaporation can partly or fully compensate for the mass gain caused by oxide growth, resulting in a very low or negative net mass change. Similar behaviour has been reported for chromia-forming alloys under humid oxidising conditions, where oxidation and chromium evaporation occur simultaneously [54]. In addition, chromium supply from the alloy bulk may become limited by diffusion, which can lead to chromium depletion near the surface and reduce the stability of the protective chromia scale [54].

Temperature also had a clear influence on oxidation behaviour. For all materials and coating systems, the mass gain was higher at 700 °C than at 600 °C. This confirms that the oxidation process is thermally activated, since higher temperatures increase reaction rates and enhance diffusion through the developing oxide scale.

Overall, the net mass-gain results show that the oxidation response was influenced by coating chemistry, coating thickness, substrate material, and temperature. Ni/Ce produced the highest mass gains, which is consistent with greater overall oxidation but may also partly reflect its larger initial coating thickness. Co/Ce showed lower and more gradual mass gain. Both coatings changed the oxidation response relative to the uncoated specimens, which were strongly affected by the balance between oxide growth and chromium evaporation. The microstructural analysis is therefore required to determine whether these differences are associated with oxide morphology, coating integrity, or the formation of protective oxide and spinel regions.

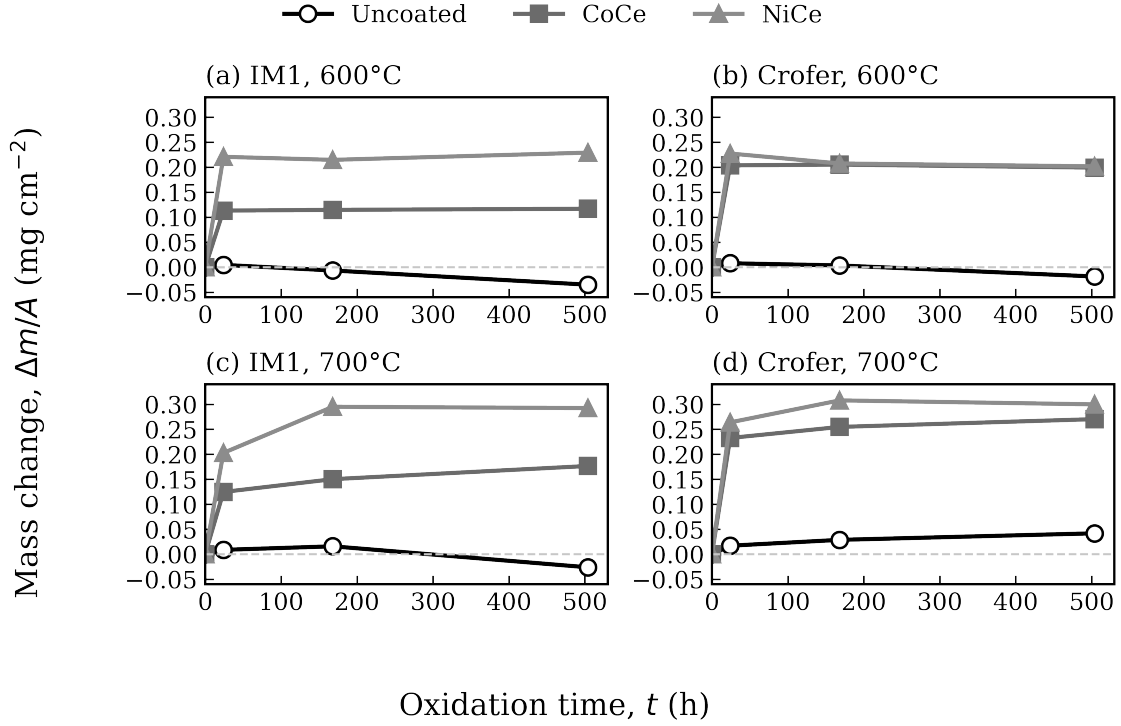


Figure 4.1: Mass change per unit area, $\Delta m/A$, as a function of oxidation time for (a) IM1 at 600 °C, (b) IM1 at 700 °C, (c) Crofer 22 APU at 600 °C, and (d) Crofer 22 APU at 700 °C, in air containing 3% H_2O .

4.1.2 Accumulated Chromium Evaporation

All investigated specimens showed an overall increase in cumulative chromium release with exposure time, as shown in Figure 4.2. This confirms that chromium volatilisation continued throughout the exposure. However, the extent of chromium release varied depending on temperature, substrate material, coating chemistry, and coating thickness.

The effect of temperature was most pronounced for the uncoated materials. When the temperature increased from 600 to 700 °C, the final cumulative chromium release increased by factors of approximately 4.5 for IM1 and 4.8 for Crofer 22 APU. This strong temperature dependence is likely related to faster chromium diffusion from the alloy bulk towards the oxide surface, together with enhanced formation of volatile chromium-containing species, mainly $\text{CrO}_2(\text{OH})_2$, in the presence of water vapour. For the coated specimens, the temperature dependence was less direct because the measured chromium release was also influenced by coating chemistry, coating thickness, and the development of protective surface oxides.

The substrate material also affected chromium release. In the uncoated condition, IM1 released approximately 1.7 times more chromium than Crofer 22 APU at 600 °C and approximately 1.6 times more at 700 °C. This indicates that Crofer 22 APU has better intrinsic resistance to chromium volatilisation. This behaviour is likely

associated with the formation of a protective outer $(\text{Mn}, \text{Cr})_3\text{O}_4$ spinel layer above the inner Cr_2O_3 scale [15]. Such a spinel layer can limit outward chromium transport and thereby reduce chromium volatilisation.

Both Co/Ce and Ni/Ce coatings reduced cumulative chromium release compared with the uncoated materials. For IM1, both coatings were effective, although their relative performance changed with temperature. At 600 °C, Ni/Ce gave the lowest chromium release, reducing the final cumulative value by a factor of approximately 5.2 compared with the uncoated specimen. Co/Ce reduced the release by a factor of approximately 4.3. At 700 °C, Co/Ce became more effective, reducing the final cumulative chromium release by a factor of approximately 8.6, compared with a reduction factor of approximately 4.1 for Ni/Ce. This shows that Co/Ce provided better chromium retention for IM1 under the higher-temperature condition.

For Crofer 22 APU, Co/Ce showed the strongest chromium-retention behaviour at both temperatures. At 600 °C, Co/Ce reduced the final cumulative chromium release by a factor of approximately 2.2, while Ni/Ce gave a reduction factor of approximately 1.6. The difference became much more pronounced at 700 °C, where Co/Ce reduced the final cumulative chromium release by a factor of approximately 20.7. In comparison, Ni/Ce reduced chromium release by a factor of approximately 3.3. This was the strongest chromium suppression observed in the present study.

The coating-thickness differences should also be considered when comparing chromium retention. The Ni/Ce coating was approximately 1000 nm thick on both substrates, while the Co/Ce coating was approximately 300 nm thick on IM1 and 600 nm thick on Crofer 22 APU. A thicker coating may provide a longer diffusion path and a larger reservoir of coating material. However, thickness alone did not determine chromium suppression. Despite being thinner than Ni/Ce, Co/Ce provided the strongest chromium retention for Crofer 22 APU and for IM1 at 700 °C. This indicates that the chemical nature, continuity, and stability of the Co-containing surface products were important, although the larger Co thickness on Crofer 22 APU may also have contributed to its particularly strong performance.

The improved performance of Co/Ce may be related to the oxidation of Co to Co_3O_4 [55], followed by the possible formation of Co-containing spinel phases such as CoCr_2O_4 and $(\text{Co}, \text{Mn})_3\text{O}_4$. Based on the literature, such phases are expected to limit outward chromium transport and reduce chromium volatilisation. The particularly strong suppression observed for Co/Ce-coated Crofer 22 APU at 700 °C may also have been supported by its approximately 600 nm Co layer, compared with approximately 300 nm on IM1. The larger Co reservoir may have promoted more complete surface coverage or sustained the formation of Co-containing oxide and spinel regions. Nevertheless, because the Ni/Ce coating was thicker at approximately 1000 nm but generally less effective at 700 °C, coating chemistry and the properties of the developed reaction products appear to have been at least as important as coating thickness.

In addition, the higher intrinsic oxidation resistance of Crofer 22 APU and its tendency to form protective outer $(\text{Mn}, \text{Cr})_3\text{O}_4$ spinel layers may have contributed to

the low chromium release. These interpretations are evaluated further using the microstructural and compositional analyses.

Ni/Ce coatings also reduced cumulative chromium release compared with the uncoated specimens, but they were generally less effective than Co/Ce, especially at 700 °C. This may be related to the formation of Ni-containing oxides and spinels, such as NiO and NiCr₂O₄, which may provide a less effective barrier to chromium transport than Co-containing phases such as Co₃O₄ and CoCr₂O₄. However, confirmation of the exact oxide phases would require complementary phase analysis. The observed trends are assessed further using microstructural characterisation.

Overall, the chromium-evaporation results show that both coatings improved chromium retention, but their effectiveness depended on substrate material, exposure temperature, coating chemistry, and coating thickness. Co/Ce gave the best overall performance, particularly for Crofer 22 APU at 700 °C, whereas Ni/Ce was most effective for IM1 at 600 °C. The performance of the thinner Co/Ce coatings demonstrates that coating thickness alone does not explain the observed chromium-retention behaviour.

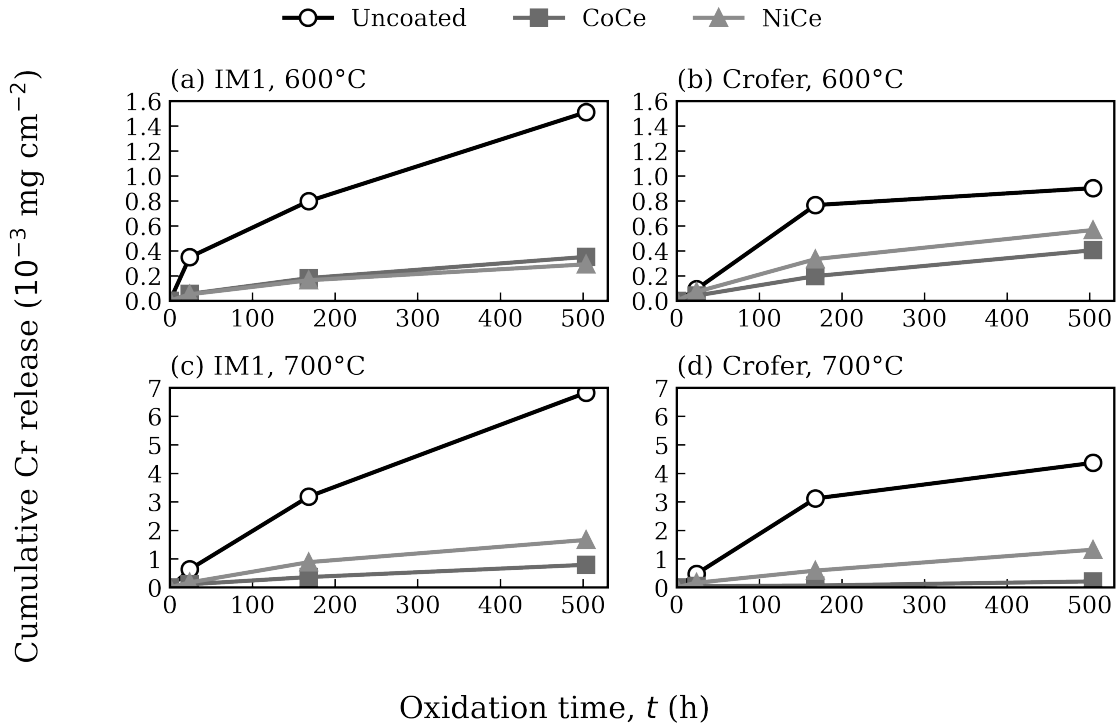


Figure 4.2: Cumulative chromium release as a function of time for (a) IM1 at 600 °C, (b) Crofer 22 APU at 600 °C, (c) IM1 at 700 °C, and (d) Crofer 22 APU at 700 °C, exposed in air containing 3% H₂O.

4.1.3 Chromium Evaporation Rate

A major advantage of the denuder technique is that the denuder tubes can be exchanged during isothermal exposure without disturbing the specimens inside the

furnace. Chromium was therefore not collected using one continuous denuder over the full exposure time. Instead, each measured value represents the amount of chromium collected during a specific exposure interval.

The interval chromium-release rates shown in Figure 4.3 complement the cumulative chromium-release results by showing how the evaporation behaviour changed with time. Although the cumulative chromium release increased throughout exposure, the interval release rates generally decreased at longer exposure times. This indicates that chromium volatilisation continued but became progressively slower as the oxide scale developed and the surface layers became more protective. The highest rates were typically observed during the first 24 h, while the lower rates measured after 168 and 504 h suggest increasing transport limitations through the growing surface scale.

The same main trends observed in the cumulative data were also reflected in the interval release rates. Higher rates were generally measured at 700 °C than at 600 °C, which is consistent with faster chromium diffusion and enhanced formation of volatile $\text{CrO}_2(\text{OH})_2$ at higher temperature. Crofer 22 APU generally showed lower rates than IM1, likely due to the formation of a protective outer $(\text{Mn}, \text{Cr})_3\text{O}_4$ spinel layer above the Cr_2O_3 scale. Among the coatings, Co/Ce gave the lowest interval chromium-release rates, especially for Crofer 22 APU at 700 °C, whereas Ni/Ce was less effective in suppressing chromium release under this condition.

The interval results should also be interpreted in the context of the unequal coating thicknesses. Although Ni/Ce had the largest initial top-layer thickness on both substrates, Co/Ce frequently produced lower chromium-release rates. This further indicates that the effectiveness of the coatings depended not only on diffusion distance, but also on the chemistry, continuity, and stability of the oxide or spinel regions formed during exposure.

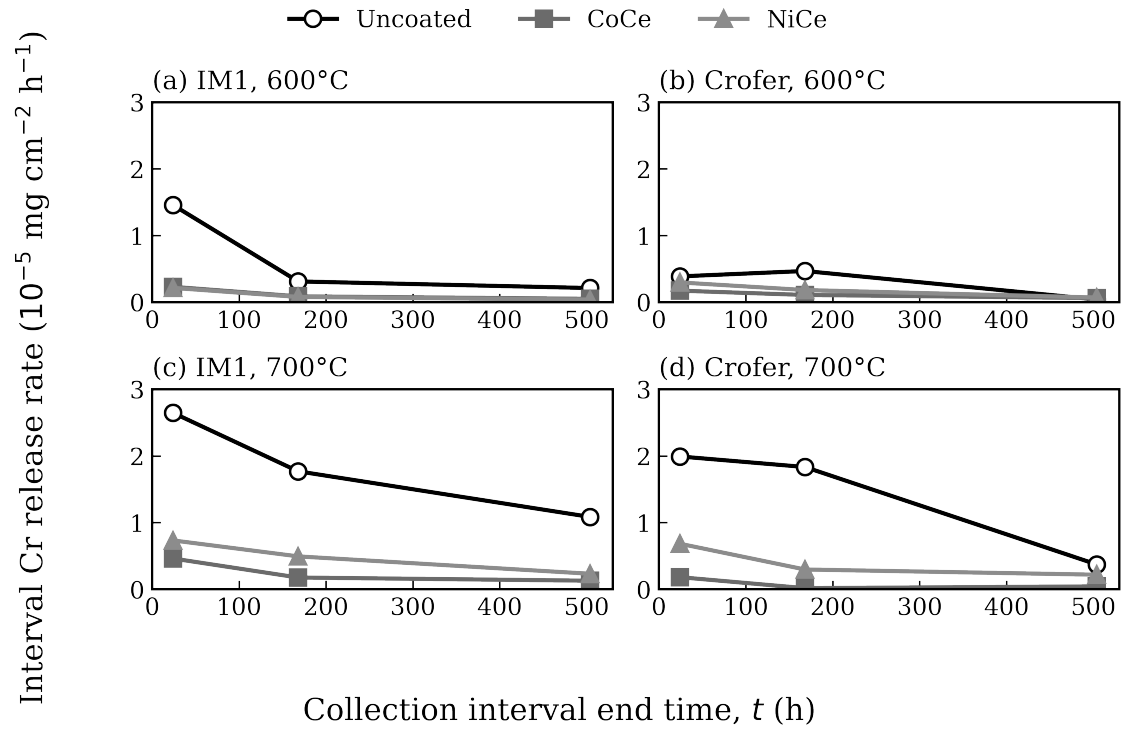


Figure 4.3: Interval chromium-release rate as a function of collection-interval end time for (a) IM1 at 600 °C, (b) Crofer 22 APU at 600 °C, (c) IM1 at 700 °C, and (d) Crofer 22 APU at 700 °C, exposed in air containing 3% H_2O .

4.1.4 Gross Mass Gain

Gross mass change was calculated because the measured mass change does not represent oxide-scale growth alone. During exposure, the specimens gain mass through oxidation but may also lose mass through chromium evaporation. The measured mass change is therefore a net value resulting from these competing processes. To better estimate the contribution from oxide formation, the evaporated chromium was converted to an equivalent amount of Cr_2O_3 and added back to the measured mass change.

As shown in Figure 4.4, the coated specimens showed positive gross mass change, whereas the uncoated specimens remained very low and, in some cases, still decreased with time. This was particularly evident for IM1. The behaviour differs from previous work, where the gross mass gain became positive after correction for chromium vaporisation, indicating that oxide-scale growth dominated after the chromium-loss correction [10]. In the present work, the Cr_2O_3 correction was not sufficient to compensate for the measured mass loss in these cases. The low or negative gross mass-change values should therefore not be interpreted as negative oxide growth, but rather as very limited net oxide formation combined with additional mass-loss effects.

Possible reasons for this behaviour include experimental scatter, since the measured

mass changes were very small, and additional mass-loss processes that are not included in the Cr_2O_3 correction. These may include minor oxide-scale spallation or loss of poorly adherent surface products. As a result, the uncoated specimens did not show clear parabolic gross mass-gain behaviour, whereas the coated specimens showed more stable positive gross mass change.

The larger gross mass gain of the Ni/Ce-coated specimens should again be interpreted in relation to their larger initial coating thickness. Since the Ni top layer was approximately 1000 nm thick, compared with 300–600 nm for the Co layers, a larger quantity of metallic coating material was available for oxidation. The gross mass-gain results therefore reflect oxidation of the complete coating/substrate system rather than only growth of an oxide scale on the steel substrate.

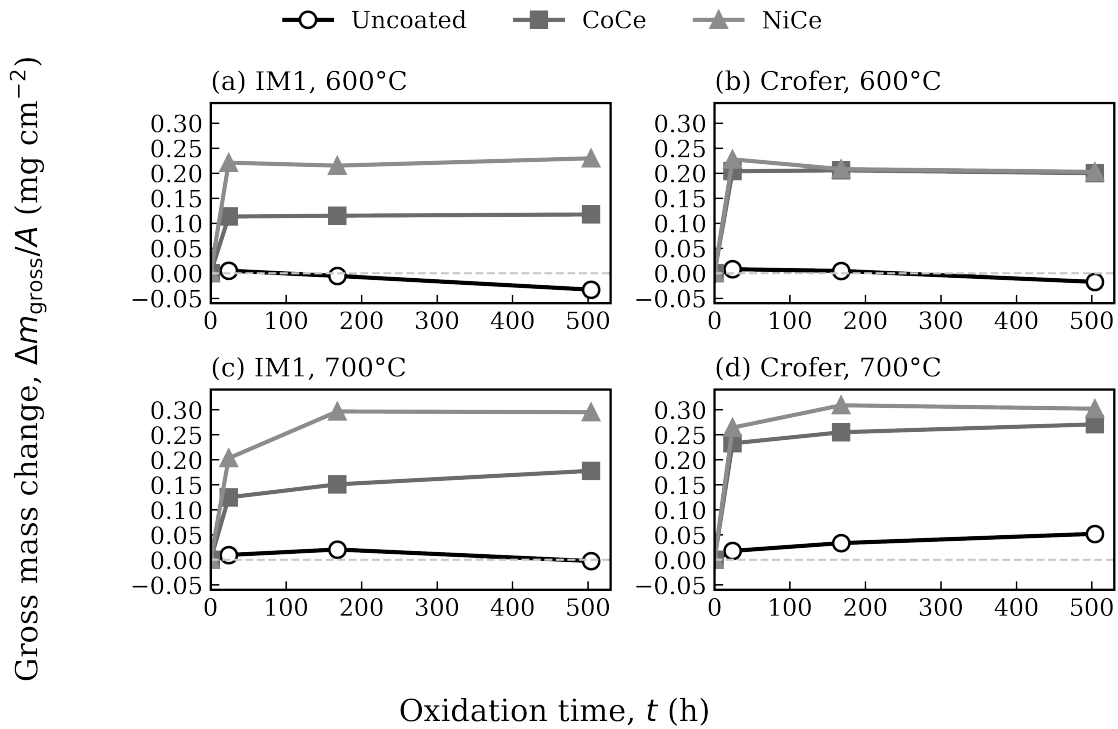


Figure 4.4: Gross mass gain per unit area, $\Delta m_{\text{gross}}/A$, corrected for chromium vaporisation, as a function of oxidation time for (a) IM1 at 600 °C, (b) Crofer 22 APU at 600 °C, (c) IM1 at 700 °C, and (d) Crofer 22 APU at 700 °C.

4.1.5 Summary of Oxidation and Chromium Evaporation Behaviour

Considering the oxidation and chromium-evaporation results together, the degradation behaviour was controlled by the combined effects of coating chemistry, coating thickness, substrate material, and temperature. Co/Ce provided the strongest overall chromium retention, particularly on Crofer 22 APU at 700 °C. Ni/Ce also reduced chromium evaporation relative to the uncoated specimens but was generally associated with higher mass gain.

The unequal coating thicknesses must be considered when interpreting these trends. The Ni/Ce coating had a 1000 nm Ni top layer on both substrates, whereas the Co/Ce coating had a 300 nm Co layer on IM1 and a 600 nm Co layer on Crofer 22 APU. The higher Ni/Ce mass gain may therefore partly reflect the larger amount of coating material available for oxidation. However, the strong chromium suppression achieved by the thinner Co/Ce coatings shows that thickness alone cannot explain the observed performance.

These results indicate that coating chemistry, coating thickness, and substrate-dependent oxide formation all contributed to the measured behaviour. The following microstructural analysis is used to examine how coating integrity, oxide morphology, and elemental distribution contributed to these differences.

4.2 Microstructural Analysis

Figure 4.6 presents backscattered-electron SEM cross-sectional images of coated and uncoated Crofer 22 APU and IM1 after 504 h of exposure at 700 °C. The images complement the gravimetric oxidation and chromium-evaporation measurements by showing the morphology and continuity of the developed oxide/coating regions.

The coated specimens in panels (A), (B), (D), and (E) generally developed comparatively continuous oxide/coating regions, whereas the corresponding uncoated specimens in panels (C) and (F) showed different oxide-scale morphologies. The oxide scale formed on Crofer 22 APU appeared more compact and uniform than that formed on IM1. This observation is consistent with the lower chromium release measured for Crofer 22 APU, although the SEM images alone do not establish oxide-scale stability or diffusion rates.

To further investigate the composition of the oxide/coating regions, EDX analyses were performed on coated and uncoated Crofer 22 APU and IM1 after 504 h of exposure at 700 °C. The EDX results complement the cross-sectional SEM observations by providing information about the presence and distribution of selected elements, including O, Cr, Fe, Mn, Co, Ni, and Ce, within the analysed regions, as shown in Figure 4.5. Together, the SEM and EDX results provide a basis for comparing oxide-scale development, elemental redistribution, and the influence of coating chemistry and substrate composition on the oxidation response.

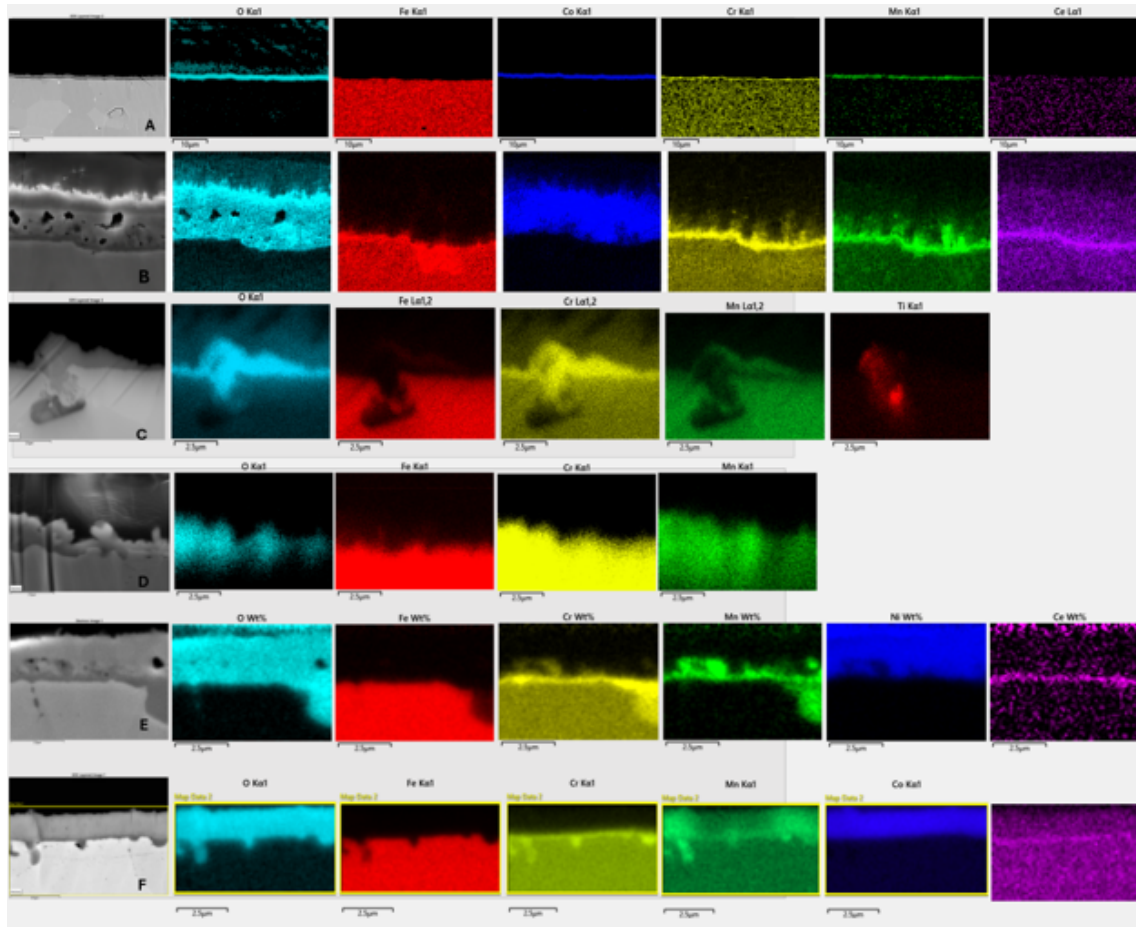


Figure 4.5: Backscattered-electron SEM cross-sectional images and corresponding EDX analyses of coated and uncoated Crofer 22 APU and IM1 after 504 h of exposure at 700 °C. Panel (A) shows Co/Ce-coated IM1, panel (B) Ni/Ce-coated IM1, panel (C) uncoated IM1, panel (D) uncoated Crofer 22 APU, panel (E) Ni/Ce-coated Crofer 22 APU, and panel (F) Co/Ce-coated Crofer 22 APU.

The Co/Ce-coated specimens generally showed relatively compact oxide/coating regions with limited visible cracking or porosity. In contrast, the Ni/Ce-coated specimens developed thicker and more compositionally complex reaction regions. These differences are consistent with the higher mass gains measured for Ni/Ce and the lower chromium-release values generally obtained for Co/Ce, particularly at 700 °C. However, the unequal initial coating thicknesses must also be considered when comparing the developed microstructures.

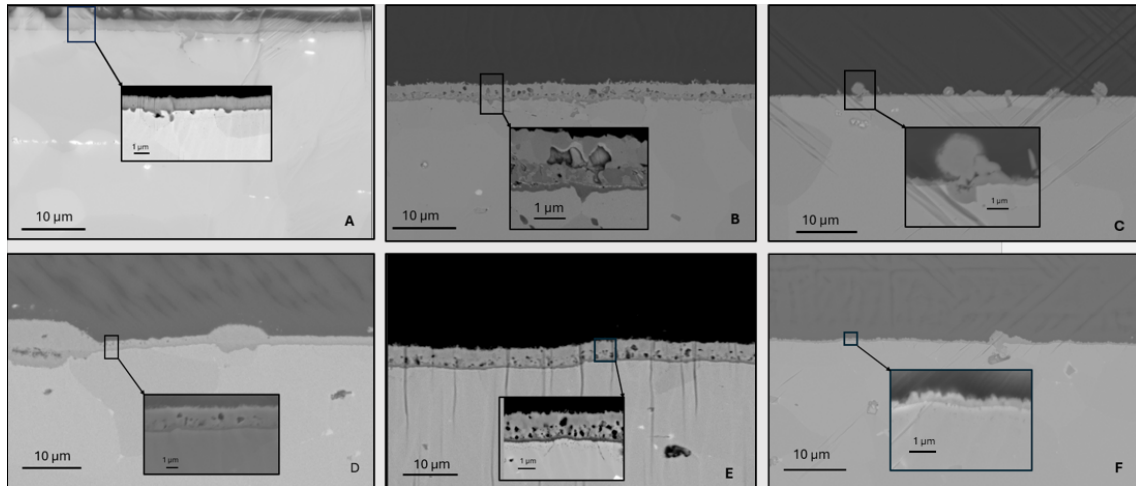


Figure 4.6: Backscattered-electron SEM cross-sectional images of coated and uncoated Crofer 22 APU and IM1 after 504 h of exposure at 700 °C. Panels (A)–(C) show Co/Ce-coated, Ni/Ce-coated, and uncoated Crofer 22 APU, respectively, while panels (D)–(F) show Co/Ce-coated, Ni/Ce-coated, and uncoated IM1, respectively.

Figure 4.7 presents backscattered-electron SEM cross-sectional images and corresponding EDX analyses of Co/Ce-coated IM1 after 504 h of exposure at 700 °C. The images show local variations in oxide-scale morphology. Isolated oxide nodules are visible in panel (C), while a larger cluster of nodules is present in panel (A). These localised regions indicate non-uniform oxide development and are consistent with local breakaway-type oxidation.

In panels (A) and (C), region (i) shows a strong Fe contribution and is therefore interpreted as an Fe-rich oxide region. Region (ii), located near the metal/oxide interface, is enriched in Cr and is consistent with the presence of an inner Cr-rich oxide layer. The available EDX data do not, however, confirm that this region consists exclusively of Cr_2O_3 .

Panel (B) shows a comparatively uniform oxide/coating region without clearly visible nodules. The corresponding EDX analysis shows a strong Co signal, confirming that coating-derived Co remained present in the developed surface region after exposure. Fe, Mn, and Cr were also detected, indicating transport of substrate elements into the growing oxide/coating region and the formation of a compositionally mixed scale.

The observed structure is consistent with oxidation mechanisms previously reported for Ce/Co-coated FSS [58]. Under oxidising conditions, the metallic Co layer is expected to convert to Co-rich oxides. Outward diffusion of Mn from the substrate may subsequently contribute to the formation of Co–Mn-containing spinel-type regions [59]. The Ce interlayer may influence oxide growth, elemental transport, and scale adhesion. Nevertheless, the exact oxide and spinel phases cannot be established from SEM/EDX alone and would require complementary phase analysis.

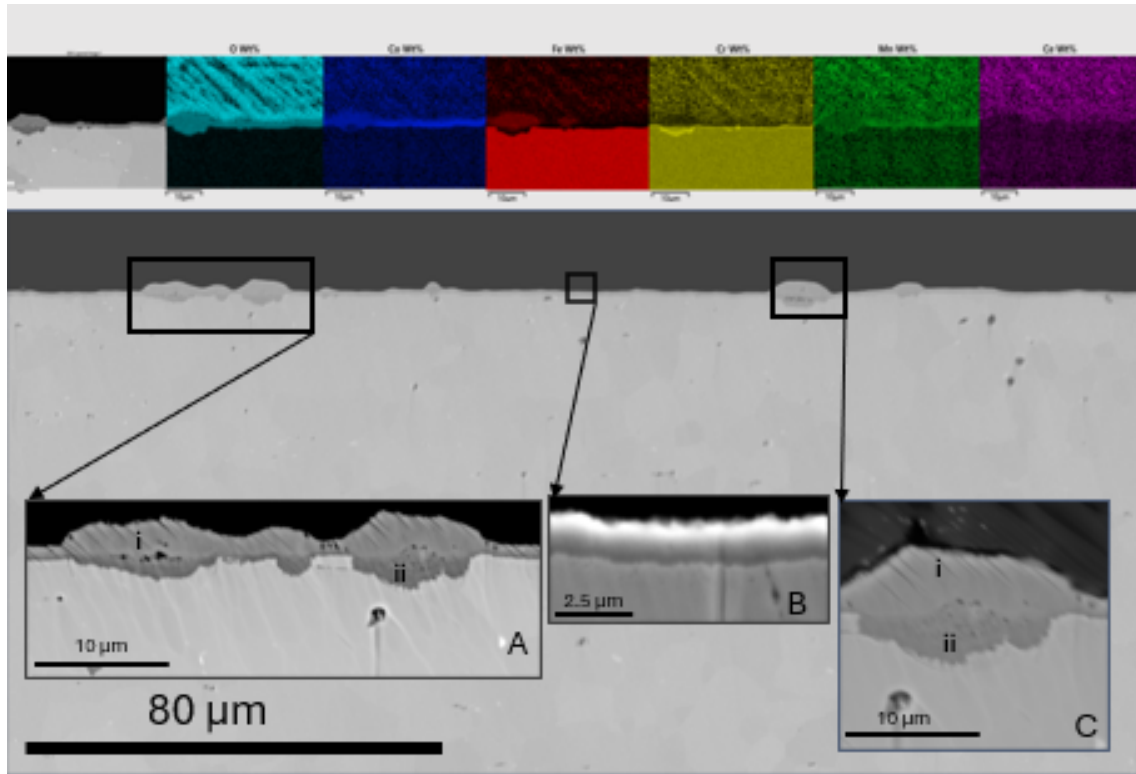


Figure 4.7: Backscattered-electron SEM cross-sectional images and corresponding EDX analyses of the oxide/coating region formed on Co/Ce-coated IM1 after 504 h of exposure at 700 °C. Isolated and clustered oxide nodules are visible in the cross sections. Region (i) is Fe-rich, while region (ii) is a Cr-rich inner oxide region located near the metal/oxide interface.

Figure 4.8 shows the oxide/coating structure developed on Ni/Ce-coated Crofer 22 APU after 504 h of exposure at 700 °C, together with the corresponding EDX elemental maps. The developed scale can be divided into three compositionally distinct regions.

Region (i) is enriched in Ni and O and is therefore interpreted as a Ni-rich outer oxide region. This distribution is consistent with the formation of a Ni-containing oxide, although the exact phase cannot be confirmed from EDX alone. Region (ii) contains contributions from several coating- and substrate-derived elements and is interpreted as an intermediate mixed-oxide region formed through outward transport of substrate elements and reaction with oxygen. Region (iii), located adjacent to the substrate, is enriched in Cr and is consistent with an inner chromia-rich oxide region.

The layered elemental distribution demonstrates that the Ni/Ce coating participated in oxide-scale development rather than remaining as an unchanged metallic layer. The presence of a comparatively thick mixed-oxide region is consistent with the higher mass gain measured for Ni/Ce-coated specimens. However, the coating still reduced chromium evaporation relative to the uncoated condition, showing that higher mass gain did not necessarily correspond to poorer chromium retention.

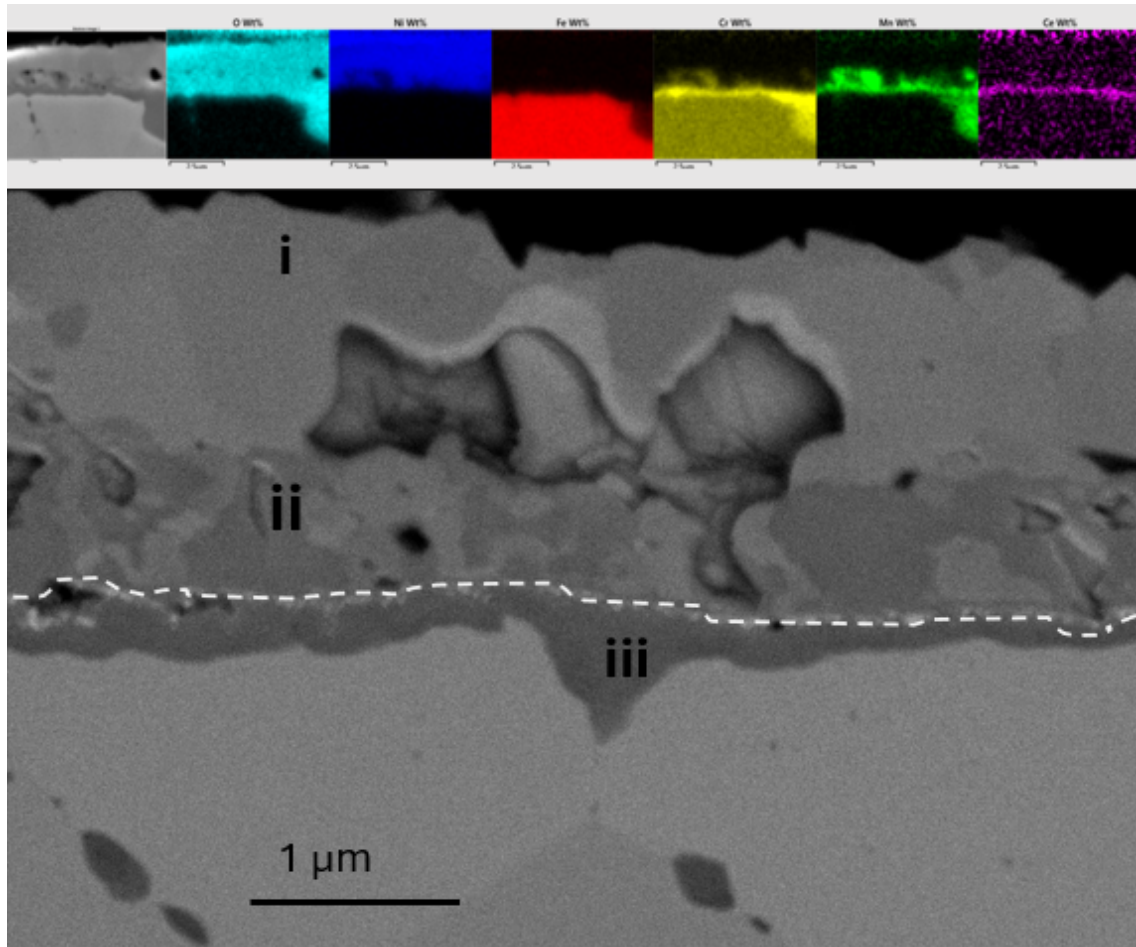


Figure 4.8: Schematic representation of the oxide/coating structure formed on Ni/Ce-coated Crofer 22 APU after 504 h of exposure at 700 °C, together with corresponding EDX elemental maps. Region (i) is a Ni-rich outer oxide region, region (ii) is an intermediate mixed-oxide region, and region (iii) is a Cr-rich inner oxide region adjacent to the FSS substrate. The dashed lines indicate approximate boundaries between the compositionally distinct regions.

Overall, the SEM and EDX results show that the coated specimens developed more compositionally complex oxide/coating structures than the uncoated materials. The Co/Ce-coated specimens generally retained Co-rich surface regions and showed comparatively compact oxide morphologies, whereas the Ni/Ce-coated specimens developed thicker mixed-oxide regions containing both coating- and substrate-derived elements.

These observations help explain why the coated specimens could show higher mass gain while still exhibiting lower chromium evaporation than the uncoated specimens. Oxidation of the metallic coating contributes to mass gain, while the developed oxide or spinel-type regions may restrict outward chromium transport. Mass gain alone is therefore insufficient for assessing coating performance and must be interpreted together with chromium-evaporation measurements, oxide-scale morphology, and elemental distribution.

The results further indicate that Co/Ce provided the more effective overall barrier under the investigated conditions. Co/Ce generally produced lower chromium-release rates and comparatively compact oxide/coating regions, whereas Ni/Ce was associated with greater mass gain and thicker mixed-oxide development. Nevertheless, the unequal initial coating thicknesses mean that the observed differences reflect the combined effects of coating chemistry and coating thickness.

4.3 Potential of IM1 as an Alternative Interconnect Material

The comparison between coated IM1 and Crofer 22 APU provides insight into the combined influence of substrate material, coating chemistry, and coating thickness. Crofer 22 APU is an established ferritic stainless steel for SOFC interconnect applications [61] and therefore provides a relevant reference material for evaluating IM1.

For the Ni/Ce-coated specimens, both IM1 and Crofer 22 APU had an initial Ni top-layer thickness of approximately 1000 nm. The two substrates showed broadly similar mass-gain and chromium-evaporation trends during exposure. The cross-sectional images obtained after 504 h at 700 °C also showed oxide/coating regions of comparable overall thickness, as presented in Figure 4.9. These observations indicate that the Ni/Ce coating provided a similar degree of protection on the two substrates under the investigated conditions.

The comparable behaviour does not imply that the substrate composition was unimportant. Differences in alloy chemistry may still influence chromium supply, interfacial reactions, oxide adhesion, and long-term coating stability. Nevertheless, the results suggest that IM1 can support a Ni/Ce coating response approaching that obtained on Crofer 22 APU.

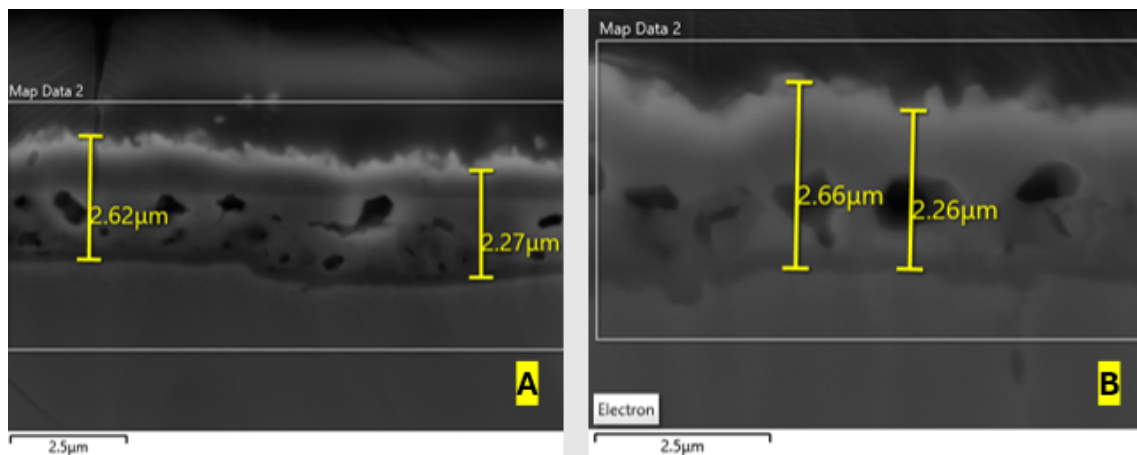


Figure 4.9: Cross-sectional images of the oxide/coating regions formed on Ni/Ce-coated (A) IM1 and (B) Crofer 22 APU after 504 h of exposure at 700 °C. Both specimens had an initial Ni top-layer thickness of approximately 1000 nm.

A similar substrate comparison can be made for the Co/Ce-coated specimens. Application of Co/Ce substantially improved the oxidation and chromium-retention behaviour of IM1 relative to the uncoated condition. The Co/Ce-coated IM1 specimens had an initial Co top-layer thickness of approximately 300 nm, whereas the corresponding coating on Crofer 22 APU contained an approximately 600 nm Co layer. Despite this twofold difference in initial Co thickness, the two coated substrates showed broadly similar mass-gain and chromium-evaporation behaviour. The thicker Co layer on Crofer 22 APU therefore did not produce a clear performance advantage under the investigated exposure conditions.

This result indicates that effective oxidation control and chromium suppression were achieved on IM1 with a lower Co loading. The comparison with Ni/Ce further shows that coating thickness alone did not determine performance. On IM1, the approximately 300 nm Co/Ce coating generally provided stronger chromium retention and lower mass gain than the approximately 1000 nm Ni/Ce coating, despite the Ni layer being more than three times thicker. Coating chemistry, phase formation, coating continuity, and interfacial stability therefore appear to have been more important than initial metallic-layer thickness alone.

The performance of the thinner Co/Ce coating is relevant from both resource-efficiency and manufacturing perspectives. Cobalt is classified by the European Union as a critical raw material owing to its economic importance and supply risk [62]. Achieving strong chromium suppression with an approximately 300 nm Co layer may reduce cobalt consumption and deposition requirements compared with a thicker Co-containing coating. If IM1 is also confirmed to be less costly than Crofer 22 APU, combining this substrate with a thinner protective coating could offer further economic advantages. However, the present study did not include a formal cost or life-cycle assessment, and these wider benefits should therefore be regarded as potential rather than quantitatively demonstrated advantages.

The Ni/Ce comparison also supports the consideration of IM1 as an alternative substrate, since the approximately 1000 nm Ni/Ce coatings behaved similarly on IM1 and Crofer 22 APU. However, because only one Ni-layer thickness was investigated, these results do not establish whether the Ni/Ce coating could be made thinner without reducing its protective performance.

Overall, the results indicate that coating design can partly compensate for differences between the two substrate materials. Ni/Ce-coated IM1 approached the behaviour of Ni/Ce-coated Crofer 22 APU, while Co/Ce-coated IM1 achieved oxidation and chromium-retention performance comparable to Co/Ce-coated Crofer 22 APU with approximately half the initial Co thickness. IM1 therefore appears to be a promising alternative substrate when combined with an appropriately designed coating system.

Further work should systematically vary the Co/Ce and Ni/Ce coating thicknesses to determine the minimum thickness required to maintain adequate oxidation resistance and chromium suppression. Longer-term exposure and complementary measurements of electrical resistance, coating adhesion, and mechanical stability are

also required before IM1 can be considered a fully validated substitute for Crofer 22 APU in practical interconnect applications.

4.4 Dual-Atmosphere Corrosion Behaviour

Dual-atmosphere exposure was used to assess the corrosion behaviour of IM1 under SOC-relevant conditions, with humid air and a hydrogen-containing atmosphere present on opposite sides of the specimens. The following subsections compare uncoated, Co/Ce-coated, and Ni/Ce-coated IM1 after exposure at 600 °C, with emphasis on visible surface changes, surface morphology and composition, and the through-thickness development of the oxide/coating regions.

4.4.1 Overview Microscopy

Figure 4.10 shows overview microscopy images of the three surface conditions after 168 and 504 h of dual-atmosphere exposure. The images reveal clear differences in surface appearance depending on coating condition and exposure time.

The uncoated IM1 specimen showed the most pronounced visible degradation. After 168 h, localised contrast variations were already present, indicating non-uniform surface oxidation. After 504 h, a larger fraction of dark and bright regions was visible, suggesting continued development of surface corrosion products.

The Co/Ce-coated specimen maintained a comparatively uniform appearance throughout the exposure. Although some surface roughening and contrast variation were visible, the change between 168 and 504 h was less pronounced than for the uncoated specimen.

The Ni/Ce-coated specimen showed a different visual response. A distinct brown/rust-coloured discolouration was visible after 168 h. After 504 h, the surface appeared more grey-toned, although local contrast variations remained. The Ni/Ce coating therefore did not maintain the same uniform visual appearance as the Co/Ce coating.

Overall, the overview images show that coating chemistry influenced the visible surface changes under dual-atmosphere conditions. The Co/Ce-coated specimen maintained the most uniform appearance, while the uncoated specimen showed more pronounced localised degradation. The Ni/Ce-coated specimen exhibited a distinct brown/rust-coloured discolouration after 168 h, followed by a more grey-toned appearance after 504 h. Since optical microscopy cannot determine the composition of the observed surface products, top-view SEM and surface EDX were used to examine the surface morphology and near-surface elemental composition after 504 h.

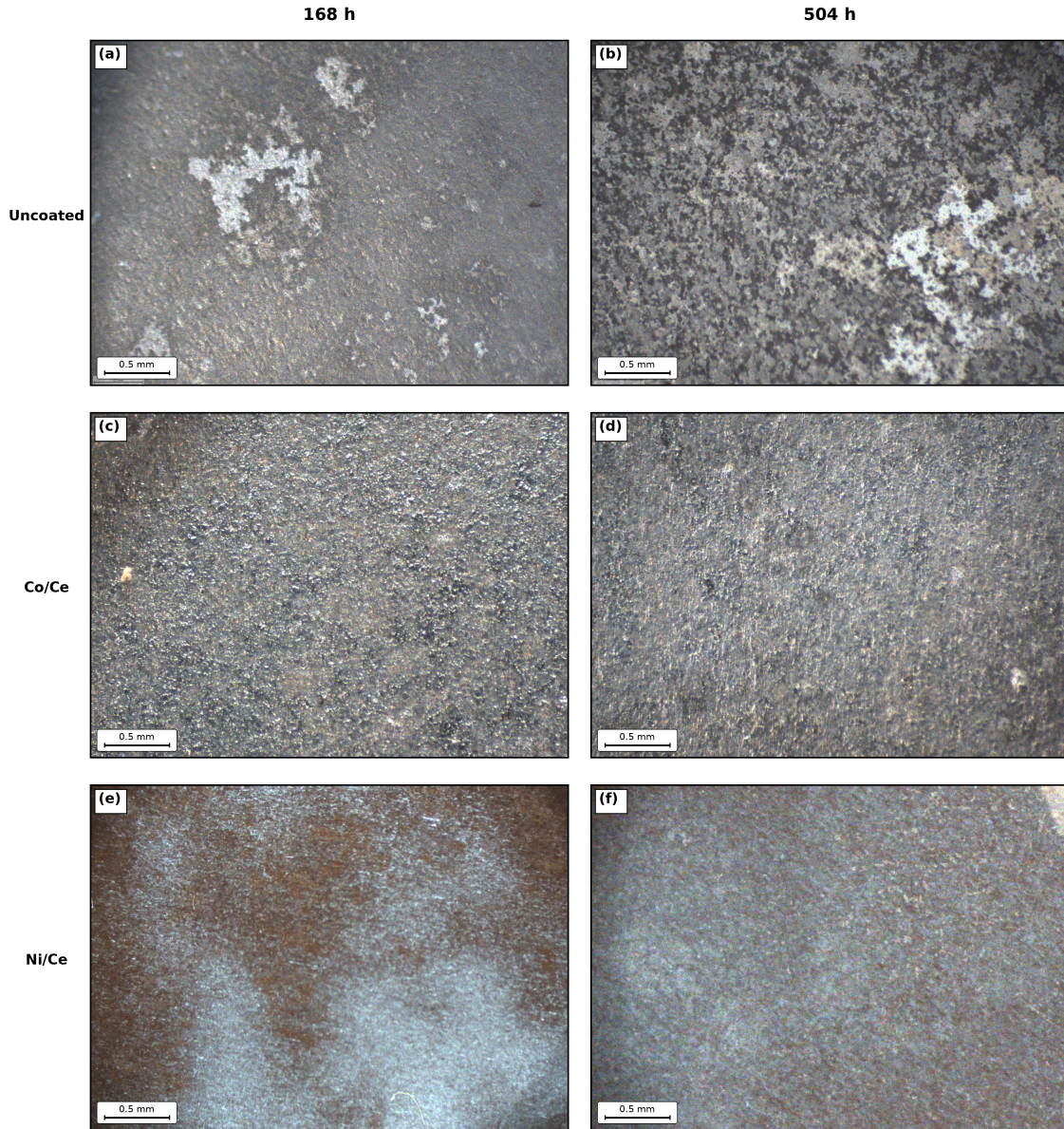


Figure 4.10: Overview microscopy images of dual-atmosphere-exposed IM1 after 168 and 504 h at 600 °C: (a,b) uncoated, (c,d) Co/Ce-coated, and (e,f) Ni/Ce-coated specimens. The images illustrate the development of visible surface changes with exposure time.

4.4.2 Top-View SEM and Surface EDX Analysis

Figure 4.11 shows top-view SEM images of the uncoated, Co/Ce-coated, and Ni/Ce-coated IM1 specimens after 504 h of dual-atmosphere exposure at 600 °C. The images are arranged from lower to higher magnification from left to right to compare the overall surface morphology and finer surface features. Surface EDX measurements provide complementary semi-quantitative information about the elemental composition of the analysed air-side regions.

The uncoated specimen showed a heterogeneous surface with densely distributed corrosion-product features and several visible cracks. At higher magnification, irregular granular and plate-like particles were observed. The cracks may have developed from stresses generated during oxide growth or cooling, although a contribution from specimen handling cannot be excluded.

Surface EDX showed that the analysed uncoated region was dominated by Fe and O, with only minor amounts of Cr and Mn. The outer surface was therefore Fe-rich rather than being dominated by a Cr-rich protective oxide. Cu was also clearly detected, indicating local enrichment at the analysed surface.

The Co/Ce-coated specimen exhibited a finer and comparatively more uniform surface morphology. Numerous small elongated or needle-like features covered the surface, while extensive cracking was less evident than for the uncoated specimen.

Surface EDX showed a substantial Co contribution together with Fe and O, confirming that coating-derived Co remained present in the outer surface region after exposure. The retained Co signal and comparatively uniform morphology indicate that the Co/Ce coating modified the development of the surface scale and maintained a coating-rich outer region.

The Ni/Ce-coated specimen also showed dense surface coverage, but its morphology was more heterogeneous than that of the Co/Ce-coated specimen. Fine particles and elongated features were observed together with larger irregular structures.

Surface EDX showed that the analysed Ni/Ce-coated region was dominated by Fe and O, while only a small amount of Ni was detected. This indicates that the final outer surface was Fe-rich and that Ni was not the dominant element in the analysed region.

The Fe-rich composition of the Ni/Ce-coated surface is consistent with Fe-containing corrosion products contributing to the brown/rust-coloured appearance observed after 168 h. However, the optical and EDX observations correspond to different exposure times, and EDX does not identify individual oxide phases. The relationship should therefore be regarded as a supported interpretation rather than direct confirmation.

Overall, the top-view SEM and surface EDX results show that the three surface conditions developed different morphologies and near-surface compositions. The uncoated specimen showed a heterogeneous, cracked, and Fe-rich surface. The Co/Ce-coated specimen exhibited a finer morphology and retained a substantial Co contribution, whereas the Ni/Ce-coated surface was more heterogeneous and strongly dominated by Fe and O. The through-thickness elemental distributions and oxide/-coating structures are examined in the following cross-sectional SEM/EDX section.

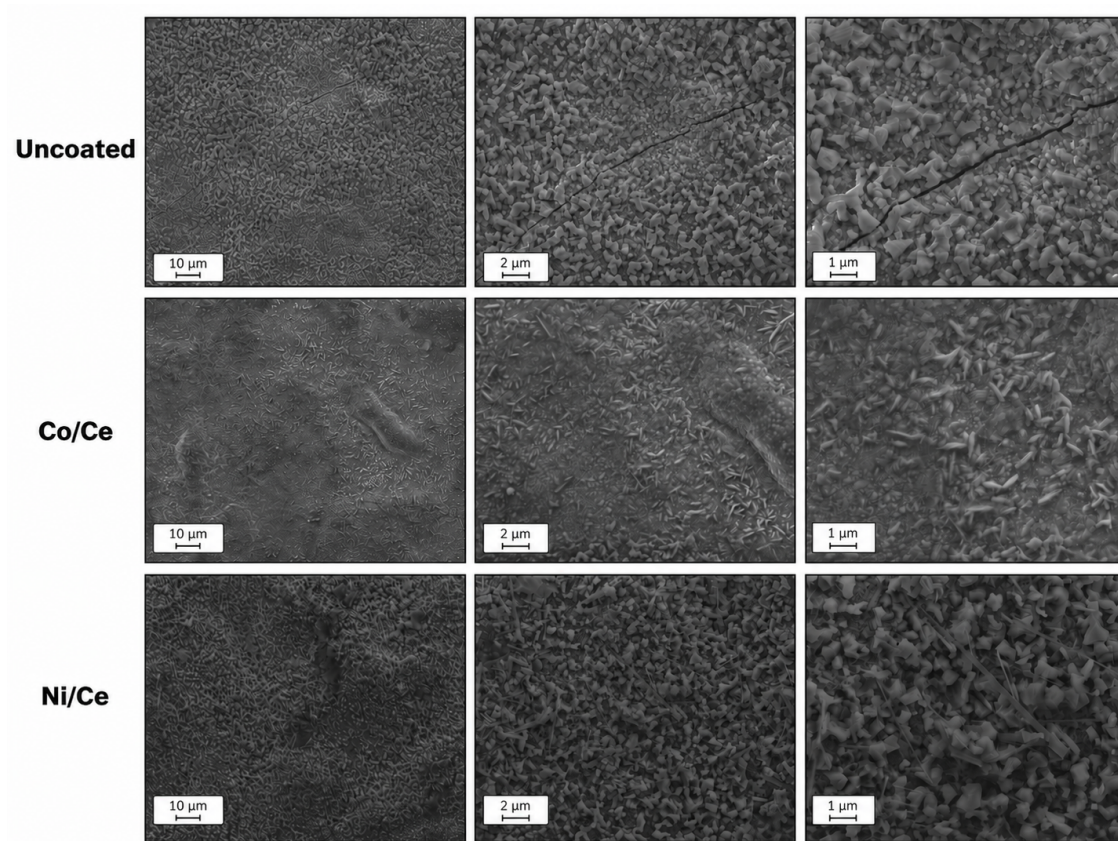


Figure 4.11: Top-view SEM images of uncoated, Co/Ce-coated, and Ni/Ce-coated IM1 after 504 h of dual-atmosphere exposure at 600 °C. Images are arranged from lower to higher magnification from left to right.

4.4.3 Cross-Sectional SEM/EDX Analysis

Figure 4.12 compares local oxide/reaction-zone thickness measurements for the uncoated, Co/Ce-coated, and Ni/Ce-coated IM1 specimens after 504 h of dual-atmosphere exposure at 600 °C. The corresponding cross-sectional SEM/EDX results are presented separately for the three surface conditions in Figures 4.13–4.15. The SEM images show the morphology of the oxide/coating regions, while the EDX maps show the spatial distributions of O and the principal alloying or coating-related elements.

Local thickness measurements indicate that the oxide/reaction zones were not uniform. The uncoated specimen showed local thicknesses of approximately 30–35 μm , while the Co/Ce-coated specimen showed lower values of approximately 18–26 μm . The Ni/Ce-coated specimen showed values of approximately 28–36 μm , indicating a comparatively thick and locally variable oxide/coating region.

Since these values were obtained from selected positions, they should be regarded as local estimates rather than statistically representative average thicknesses. Nevertheless, the measurements indicate clear differences in oxide-scale development among the three surface conditions.

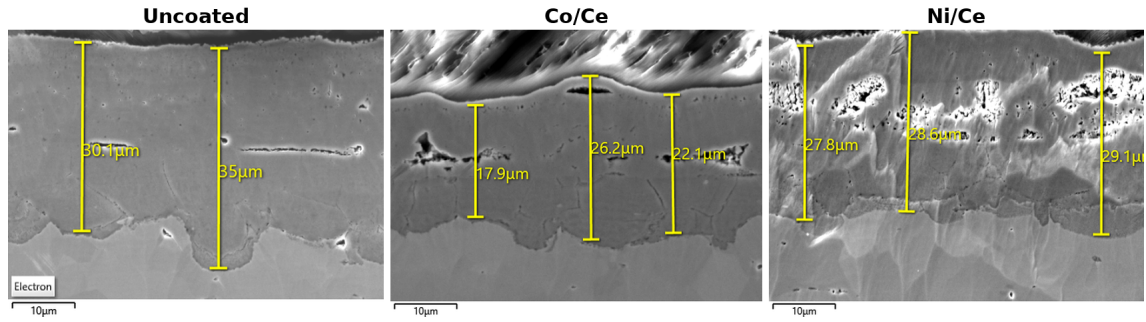


Figure 4.12: Cross-sectional SEM images showing local oxide/reaction-zone thickness measurements for uncoated, Co/Ce-coated, and Ni/Ce-coated IM1 after 504 h of dual-atmosphere exposure at 600 °C. The indicated values represent local measurements and should not be interpreted as statistically averaged oxide-scale thicknesses.

For the uncoated IM1 specimen, Figure 4.13 shows oxygen enrichment throughout the surface reaction zone, confirming extensive oxide formation. Fe is detected through a large part of the oxide region, whereas Cr is more strongly associated with the inner part of the scale. The different Fe and Cr distributions indicate that the scale was not composed solely of a thin, uniformly Cr-rich protective oxide. Instead, the maps are consistent with a chemically layered scale containing a more Fe-rich outer region and a Cr-containing inner region.

Mn largely follows the Cr-rich region, suggesting that Mn is associated with the Cr-containing oxide. Cu is concentrated mainly towards the outer part of the scale, despite the relatively low nominal Cu content of IM1.

This elemental distribution is consistent with previous observations of dual-atmosphere corrosion of AISI 441 at 600 °C. Alnegren et al. reported breakdown of protective oxidation and the development of Fe-rich corrosion products under dual-atmosphere conditions, whereas specimens exposed to humid air on both sides maintained thinner protective chromia-based scales [17]. The Fe-rich character of the scale observed in the present work therefore indicates that oxidation was not limited to stable growth of a thin protective chromia layer.

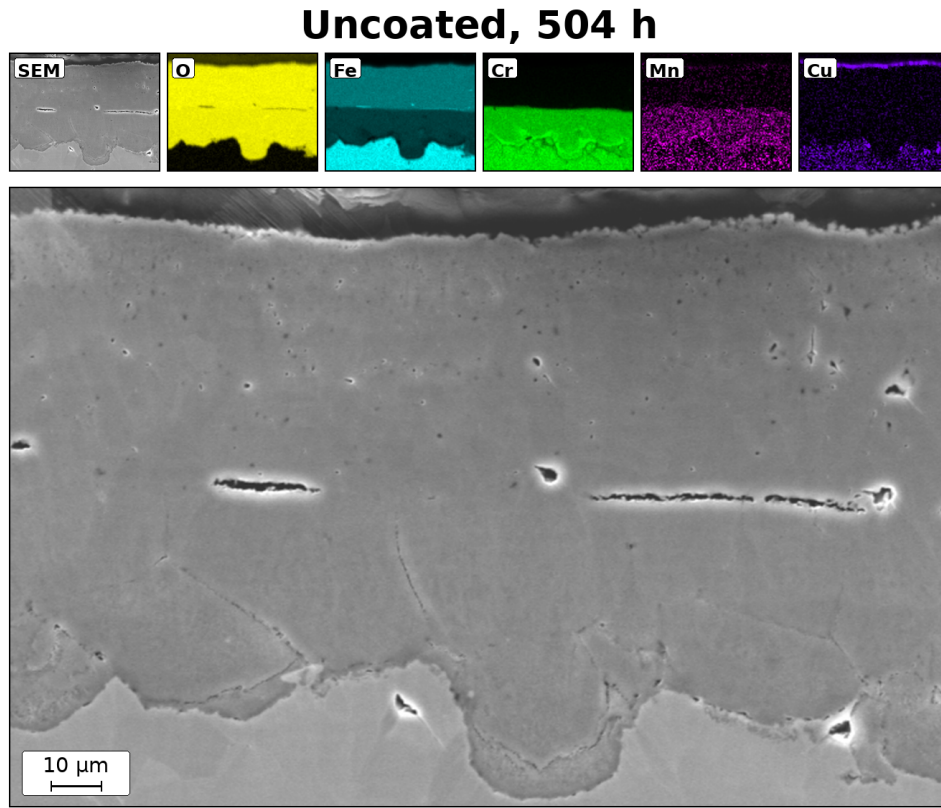


Figure 4.13: Cross-sectional SEM image and corresponding EDX elemental maps of uncoated IM1 after 504 h of dual-atmosphere exposure at 600 °C. The maps show the distributions of O, Fe, Cr, Mn, and Cu across the oxide scale and near-surface region.

The Co/Ce-coated specimen showed a different oxide/coating structure, as shown in Figure 4.14. Co is concentrated mainly in the outer surface region, while Fe is located primarily beneath the Co-rich region and is not clearly detected above it. This indicates that the Co-containing outer layer remained compositionally distinguishable from the underlying Fe-containing oxide and substrate. The observed separation suggests that the Co/Ce coating restricted outward Fe transport during exposure.

Cr and Mn are concentrated closer to the inner oxide region and show similar spatial distributions. Their overlap is consistent with the presence of a Cr–Mn-containing oxide region. Cu is most visible near the outer part of the scale.

Together with its comparatively lower local reaction-zone thickness, the limited Fe signal above the Co-rich region suggests that the Co/Ce coating restricted outward Fe transport and oxide-scale development more effectively than the Ni/Ce coating under the present conditions. However, the EDX maps are qualitative and do not by themselves establish exact oxide phases or diffusion rates.

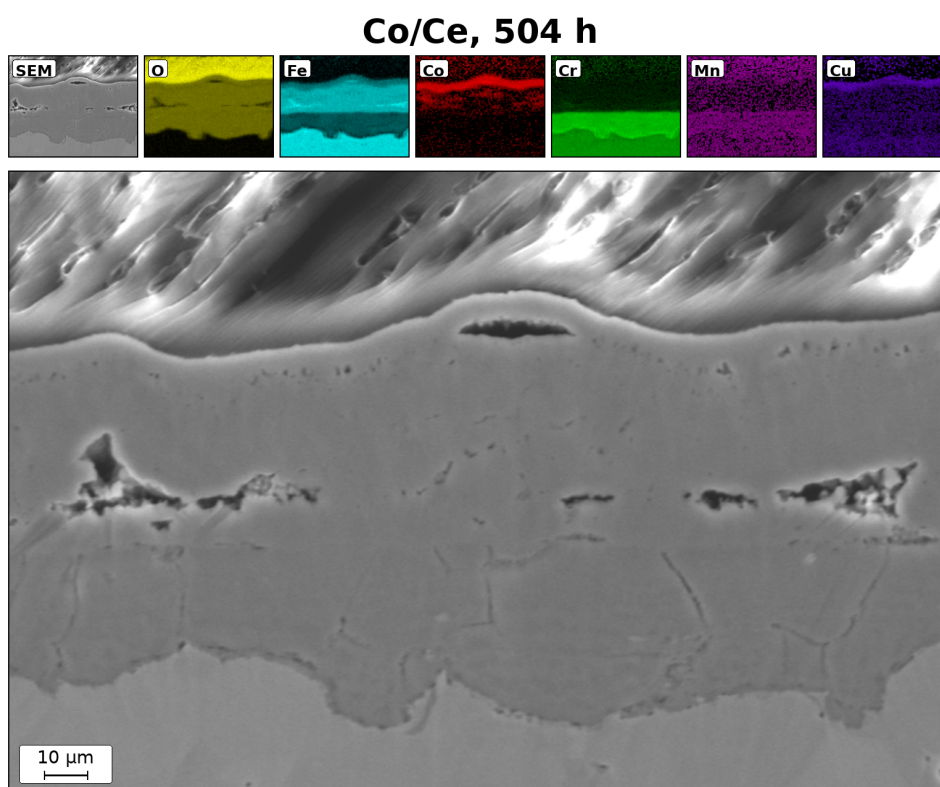


Figure 4.14: Cross-sectional SEM image and corresponding EDX elemental maps of Co/Ce-coated IM1 after 504 h of dual-atmosphere exposure at 600 °C. The maps show the distributions of O, Fe, Co, Cr, Mn, and Cu across the oxide/coating region and near-surface region.

In contrast, the Ni/Ce-coated specimen in Figure 4.15 shows Fe extending through much of the oxide/coating region. This distribution is consistent with outward Fe transport from the substrate and indicates that Fe participated strongly in the growing surface scale. Ni remains detectable in the outer surface region, showing that coating-derived Ni was retained in or contributed to the oxide/coating layer. However, the penetration of the Fe signal through the reaction zone suggests that the Ni/Ce coating did not restrict outward Fe transport as effectively as the Co/Ce coating.

The comparatively thick and locally variable reaction zone, together with the Fe signal extending through much of the oxide/coating region, indicates the development of a compositionally mixed Fe-containing scale. Cr and Mn again show similar distributions within the inner part of the reaction zone, which is consistent with their association in a Cr–Mn-containing oxide region. Cu is visibly enriched towards the outer part of the scale, despite its low concentration in the bulk alloy.

The extensive Fe distribution in the Ni/Ce-coated cross section is also consistent with the Fe-rich surface composition identified by surface EDX.

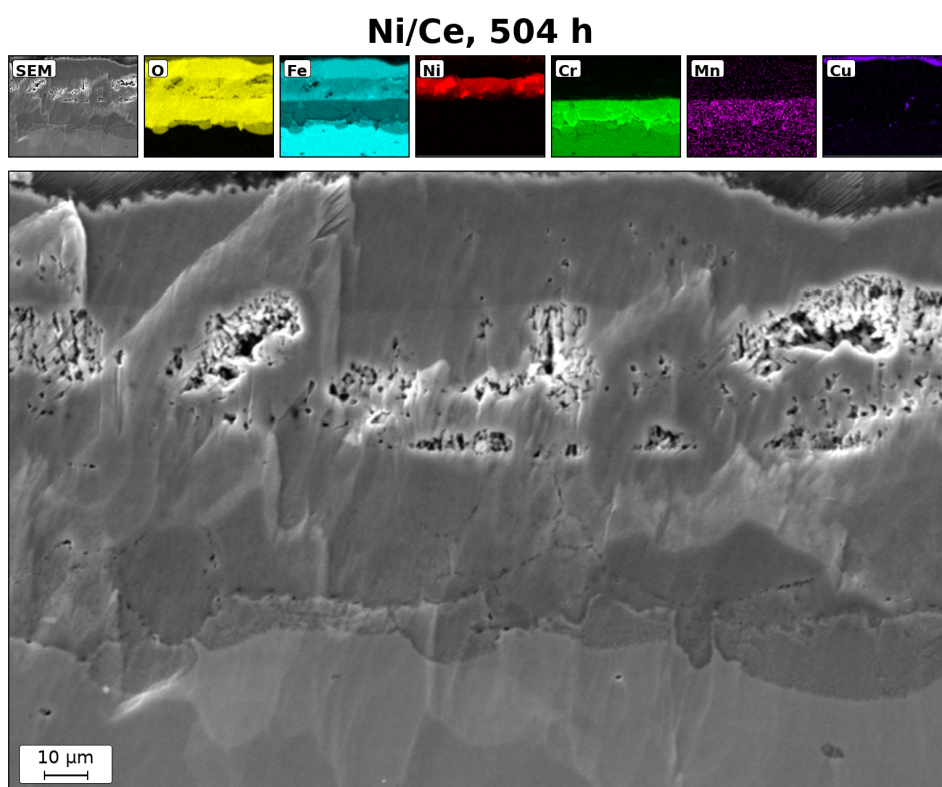


Figure 4.15: Cross-sectional SEM image and corresponding EDX elemental maps of Ni/Ce-coated IM1 after 504 h of dual-atmosphere exposure at 600 °C. The maps show the distributions of O, Fe, Ni, Cr, Mn, and Cu across the oxide/coating region and near-surface region.

Across all three specimens, Mn largely coincides with the Cr-containing regions. In FSS, Mn may become incorporated into Cr-containing spinel-type oxides. The observed Mn–Cr overlap is therefore consistent with the presence of a mixed Cr–Mn-containing oxide. Nevertheless, EDX identifies elemental distributions rather than crystalline phases, and confirmation of specific phases such as $(\text{Cr}, \text{Mn})_3\text{O}_4$ would require complementary phase analysis.

The Cu distribution is also notable because IM1 contains only a relatively small amount of Cu. Its visibility near the outer surface suggests that Cu became locally enriched during oxidation rather than remaining uniformly distributed throughout the substrate. This may be associated with outward transport, rejection from the growing Cr-rich inner oxide, or segregation within the outer oxide/coating region. The available EDX maps do not determine the chemical state of Cu, and its role in the corrosion mechanism therefore remains uncertain.

The measured oxide/reaction-zone thicknesses are comparable to literature observations for AISI 441 exposed under dual-atmosphere conditions at 600 °C. Alnegren et al. [57] reported approximately 25 μm -thick $\text{Fe}_2\text{O}_3/(\text{Fe}, \text{Cr})_3\text{O}_4$ -type scales on substantial parts of the air-facing surface under dual-atmosphere exposure, whereas

reference specimens exposed to humid air on both sides formed thin protective $(\text{Cr, Mn})_3\text{O}_4/\text{Cr}_2\text{O}_3$ scales.

The relatively thick reaction zones and substantial Fe involvement observed in the present study are therefore consistent with dual-atmosphere-assisted degradation rather than only the continued growth of a thin protective chromia-based scale.

Overall, the cross-sectional SEM/EDX analysis revealed clear differences in oxide-scale development among the three surface conditions. The uncoated specimen formed a relatively thick Fe-containing scale with Cr and Mn concentrated more strongly towards the inner region. The Co/Ce-coated specimen showed the lowest local reaction-zone thickness and retained a compositionally distinct Co-rich outer region above the Fe-containing oxide. In contrast, Fe extended through much of the thicker Ni/Ce oxide/coating region. These observations indicate that Co/Ce restricted outward Fe transport and oxide-scale growth more effectively than Ni/Ce under the investigated dual-atmosphere conditions.

5

Conclusion

This thesis investigated the influence of substrate material, coating chemistry, coating thickness, temperature, and exposure environment on the durability of ferritic stainless-steel interconnects for solid oxide electrolysis cell applications. Co/Ce and Ni/Ce coatings were evaluated on Crofer 22 APU and IM1 during exposure in humid air at 600 and 700 °C for up to 504 h, while dual-atmosphere exposures were performed on IM1 at 600 °C. The coating systems were assessed using mass-change measurements, chromium-evaporation measurements, and surface and cross-sectional SEM/EDX analyses.

Both Co/Ce and Ni/Ce substantially reduced chromium evaporation compared with the corresponding uncoated materials, demonstrating their ability to restrict chromium transport to the gas phase. Ni/Ce produced the lowest cumulative chromium release for IM1 at 600 °C, showing that a cobalt-free coating can provide effective chromium suppression under selected conditions. However, Co/Ce provided the strongest overall chromium retention, particularly at 700 °C. The greatest improvement was observed for Co/Ce-coated Crofer 22 APU at 700 °C, for which the cumulative chromium release after 504 h was approximately 20.7 times lower than for the uncoated material.

The coated specimens generally exhibited higher positive mass gains than the uncoated materials. Ni/Ce showed the highest mass gains, whereas Co/Ce exhibited lower and more gradual mass increases. Since the initial coating thicknesses differed, the greater mass gain of Ni/Ce was likely influenced by the larger amount of coating material available for oxidation. Nevertheless, the stronger chromium suppression achieved by the thinner Co/Ce coatings demonstrates that coating thickness alone did not determine protective performance. The approximately 300 nm Co/Ce coating on IM1 generally provided stronger chromium retention and lower mass gain than the approximately 1000 nm Ni/Ce coating on the same substrate. This finding indicates that increasing the initial metallic-layer thickness did not necessarily improve protection under the investigated conditions and highlights the importance of coating chemistry and the resulting oxide/coating structure.

Crofer 22 APU exhibited better intrinsic resistance to chromium evaporation than IM1 in the uncoated condition, but this difference was substantially reduced by the protective coatings. Ni/Ce-coated IM1 and Crofer 22 APU showed broadly similar oxidation behaviour, chromium-evaporation trends, and oxide-scale development. Co/Ce-coated IM1 also achieved mass-gain and chromium-retention behaviour approaching that of Co/Ce-coated Crofer 22 APU despite having approximately half the initial Co-layer thickness. These findings indicate that IM1 can approach the performance of Crofer 22 APU when protected by a suitable coating. More generally, the results show that the observed behaviour was governed by the combined effects of coating chemistry, coating thickness, and substrate composition.

The microstructural observations supported the oxidation and chromium-evaporation results. The coated specimens generally developed more continuous oxide/coating regions than the uncoated materials, while the final scale morphology depended on both coating chemistry and substrate composition. Under dual-atmosphere conditions, Co/Ce-coated IM1 exhibited the lowest local reaction-zone thickness and retained a compositionally distinct Co-rich outer region. In contrast, Ni/Ce-coated IM1 developed a thicker and more heterogeneous oxide/coating region, with Fe extending through much of the reaction zone. These observations indicate that Co/Ce restricted outward Fe transport and oxide-scale development more effectively than Ni/Ce under the investigated dual-atmosphere conditions.

Overall, Ni/Ce demonstrated potential as a cobalt-free coating alternative because it reduced chromium evaporation on both substrates and performed particularly well on IM1 at 600 °C. However, it did not provide the same overall level of protection as Co/Ce, particularly at 700 °C and under dual-atmosphere conditions. Ni/Ce should therefore be regarded as a promising but not yet equivalent replacement for Co/Ce. Co/Ce provided the most robust overall protection, while IM1 showed potential as an alternative interconnect substrate when combined with a suitable coating system.

Further work should systematically investigate the influence of coating thickness and determine the minimum thickness required to maintain effective oxidation resistance and chromium suppression. In particular, thinner Ni/Ce coatings should be evaluated to determine whether the current 1000 nm layer can be reduced without further loss of protection. Longer exposure times, repeated dual-atmosphere testing, and complementary measurements of electrical resistance, coating adhesion, mechanical stability, and phase composition are also required to assess the long-term technical and economic suitability of IM1 and Ni/Ce for practical SOEC interconnect applications.

Bibliography

- [1] IPCC (2023). *Climate Change 2023: Synthesis Report*. Intergovernmental Panel on Climate Change. <https://www.ipcc.ch/report/ar6/syr/>
- [2] International Energy Agency (2024). *CO₂ Emissions in 2023*. IEA, Paris. <https://www.iea.org/reports/co2-emissions-in-2023>
- [3] Lund, H., et al. (2017). Energy storage and smart energy systems. *Energy*, 91, 631–646. <https://doi.org/10.1016/j.energy.2017.05.123>
- [4] International Energy Agency (2019). *The Future of Hydrogen*. IEA, Paris. <https://www.iea.org/reports/the-future-of-hydrogen>
- [5] International Energy Agency (2025). *Global Hydrogen Review 2025*. IEA, Paris. <https://www.iea.org/reports/global-hydrogen-review-2025>
- [6] Laguna-Bercero, M. A. (2012). *Recent advances in high temperature electrolysis using solid oxide fuel cells: A review*. *Journal of Power Sources*, 203, 4–16. <https://doi.org/10.1016/j.jpowsour.2011.12.019>
- [7] Sun, J., Ebbesen, S. D., Mogensen, M. B. (2011) Solid oxide electrolysis cells: materials and challenges, *Energy & Environmental Science*, 4, 3805–3819.
- [8] Fergus, J. W. (2005). Metallic interconnects for solid oxide fuel cells. *Materials Science and Engineering A*, 397, 271–283. <https://doi.org/10.1016/j.msea.2005.02.047>
- [9] Zhu, W. Z., Deevi, S. C. (2003). Development of interconnect materials for solid oxide fuel cells. *Materials Science and Engineering A*, 348, 227–243. <https://www.sciencedirect.com/science/article/pii/S0921509302007360>
- [10] Falk-Windisch, H., Svensson, J.-E., Froitzheim, J. (2015). The effect of water vapor on chromium vaporization and oxide scale growth. *Journal of Power Sources*, 287, 25–35. <https://doi.org/10.1016/j.jpowsour.2015.04.040>
- [11] Fergus, J. W. (2007). Chromium poisoning in SOFC cathodes. *International Journal of Hydrogen Energy*, 32, 3664–3671. <https://doi.org/10.1016/j.ijhydene.2006.08.005>

-
- [12] Reddy et al. (2023). Experimental review of the performances of protective coatings for interconnects in solid oxide fuel cells. *Journal of Power Sources*, 568, 232831. <https://doi.org/10.1016/j.jpowsour.2023.232831>
- [13] Hilger et al. (2025). Towards sustainable interconnects for solid oxide cells: An integrated technical and environmental evaluation of coating methods. *Journal of Power Sources*, 659, 238471. <https://doi.org/10.1016/j.jpowsour.2025.238471>
- [14] Reddy et al. (2026). Comparison of Ce/Co and Ce/FeNi coatings for interconnects in solid oxide electrolysis. *International Journal of Hydrogen Energy*, 203, 152996. <https://doi.org/10.1016/j.ijhydene.2025.152996>
- [15] Sachitanand, R., Sattari, M., Svensson, J.-E. and Froitzheim, J. (2013). Evaluation of the oxidation and Cr evaporation properties of selected FeCr alloys used as SOFC interconnects. *International Journal of Hydrogen Energy*, 38(35), 15328–15334. <https://doi.org/10.1016/j.ijhydene.2013.09.044>
- [16] Reddy, M. J., Chausson, T. E., Svensson, J.-E. and Froitzheim, J. (2023). 11–23% Cr steels for solid oxide fuel cell interconnect applications at 800 °C – How the coating determines oxidation kinetics. *International Journal of Hydrogen Energy*, 48(34), 12893–12904. <https://doi.org/10.1016/j.ijhydene.2022.11.326>
- [17] Alnegren, P., Sattari, M., Svensson, J.-E., and Froitzheim, J. (2016). Severe dual atmosphere effect at 600 °C for stainless steel 441. *Journal of Power Sources*. <https://doi.org/10.1016/j.jpowsour.2015.10.001>
- [18] Solid Oxide Electrolyser Cell, *ScienceDirect Topics*, Elsevier. Available at: <https://www.sciencedirect.com/topics/engineering/solid-oxide-electrolyser-cell> (Accessed: 18 May 2026).
- [19] Küngas, R. (2020) Review—Electrochemical CO₂ Reduction for CO Production: Comparison of Low- and High-Temperature Electrolysis Technologies, *Journal of The Electrochemical Society*, 167, 044508.
- [20] Tomas, M. (2024) Corrosion of Metallic Interconnects for Solid Oxide Fuel Cells: Mitigating Chromium Evaporation and Dual-Atmosphere Challenges, PhD Thesis, Chalmers University of Technology.
- [21] Mogensen, M. B. (2020) Thermodynamics of high temperature H₂O and CO₂ electrolysis, *Figshare*, doi:10.6084/m9.figshare.12652322.v, <https://doi.org/10.6084/m9.figshare.12652322.v>.
- [22] Scott, K. (2019) Introduction to Electrolysis, Electrolysers and Hydrogen Production, in K. Scott (Ed.), *Electrochemical Methods for Hydrogen Production*, The Royal Society of Chemistry. DOI: 10.1039/9781788016049-00001.
- [23] Singhal, S. C., Kendall, K. (2003). *High Temperature Solid Oxide Fuel Cells*. Elsevier. <https://www.sciencedirect.com/book/9781856173872>

-
- [24] Lehner, M., Tichler, R., Steinmüller, H., Koppe, M. (2014) *Power-to-Gas: Technology and Business Models*, Springer.
- [25] O'Brien, J. E., McKellar, M. G., Hawkes, G. L. (2020). High-temperature electrolysis for large-scale hydrogen production. *Journal of The Electrochemical Society*, 167(5), 054511. <https://doi.org/10.1016/j.ijhydene.2008.12.021>
- [26] Wang, Y., Liu, T., Lei, L., Chen, F. (2017) High temperature solid oxide H₂O/CO₂ co-electrolysis for syngas production, *Fuel Processing Technology*, 161, 47–55.
- [27] Kreuer, K.D. (2003) Proton-conducting oxides, *Annual Review of Materials Research*, 33, 333–359.
- [28] Laguna-Bercero, M.A., Wang, Y., Zhou, X.-D., Zhu, L. (2023) Fundamentals of Solid Oxide Electrolysis Cells (SOEC), in: *High Temperature Electrolysis*, Lecture Notes in Energy, vol. 95, Springer, Cham, pp. 5–34, doi:10.1007/978-3-031-22508-6_2.
- [29] Wang, Y., Liu, T., Lei, L., Chen, F. (2017) High temperature solid oxide H₂O/CO₂ co-electrolysis for syngas production, *Fuel Processing Technology*, 161, 248–258, doi:10.1016/j.fuproc.2016.08.009.
- [30] Doenitz, W., Schmidberger, R., Steinheil, E., Streicher, R. (1980) Hydrogen production by high temperature electrolysis of water vapour, *International Journal of Hydrogen Energy*, 5(1), 55–63.
- [31] Eguchi, K., Hatagishi, T., Arai, H. (1996) Power generation and steam electrolysis characteristics of an electrochemical cell with a zirconia- or ceria-based electrolyte, *Solid State Ionics*, 86–88, 1245–1249.
- [32] Ni, M., Leung, M. K. H., Leung, D. Y. C. (2010) Technological development of hydrogen production by solid oxide electrolyzer cell (SOEC), *International Journal of Hydrogen Energy*, 35, 518–528. <https://doi.org/10.1016/j.ijhydene.2009.11.081>.
- [33] Ji, Y., Kilner, J. A., Carolan, M. F. (2005) Electrical properties and oxygen diffusion in yttria-stabilised zirconia (YSZ)–La_{0.8}Sr_{0.2}MnO_{3±δ} (LSM) composites, *Solid State Ionics*, 176(9), 937–943. <https://doi.org/10.1016/j.ssi.2004.11.019>.
- [34] Ostergard, M.J.L., Clausen, C., Bagger, C., Mogensen, M. (1995) Manganite-zirconia composite cathodes for SOFC: influence of structure and composition, *Electrochimica Acta*, 40(12), 1971–1981.
- [35] Mogensen, M., Primdahl, S., Jorgensen, M.J., Bagger, C. (2000) Composite electrodes in solid oxide fuel cells and similar solid state devices, *Journal of Electroceramics*, 5(2), 141–152.

-
- [36] Takahashi, T., & Nguyen, Q. M. (1995). Science and Technology of Ceramic Fuel Cells. *Elsevier, 1st edition*, p. 165.
- [37] Mah, J. C. W., Muchtar, A., Somalu, M. R., & Ghazali, M. J. (2017). Metallic interconnects for solid oxide fuel cells: A review on protective coating and deposition techniques. *International Journal of Hydrogen Energy*, 42(14), 9219–9229. <https://doi.org/10.1016/j.ijhydene.2017.02.026>
- [38] Goebel, C. (2020). *Strategies to Mitigate the Degradation of Stainless-Steel Interconnects Used in Solid Oxide Fuel Cells*. Doctoral thesis, Chalmers University of Technology. ISBN 978-91-7905-358-1. https://research.chalmers.se/publication/519222/file/519222_Fulltext.pdf
- [39] Kofstad, P. (1988). *High Temperature Corrosion*. Elsevier Applied Science, London. <https://archive.org/details/hightemperaturec0000kofs>
- [40] Young, D. J. (2016). “Enabling Theory,” in *High Temperature Oxidation and Corrosion of Metals*, Elsevier, pp. 29–79. <https://www.sciencedirect.com/science/chapter/monograph/pii/B9780081001011000029>
- [41] Ellingham, H. J. T. (1944). Reducibility of oxides and sulphides in metallurgical processes. *Journal of the Society of Chemical Industry*, 63(5), 125–160. <https://onlinelibrary.wiley.com/doi/epdf/10.1002/jctb.5000630501>
- [42] Northwestern University, Materials Science and Engineering Core. Ellingham diagram. Available at: https://msecore.northwestern.edu/315/12_home_ken_Mydocs_MSEcore_315_figures_Ellingham-diagram.svg
- [43] Sand, K., Falk-Windisch, H., Svensson, J.-E., Froitzheim, J. (2019). Chromium vaporization from chromia-forming alloys in humid atmospheres. Chalmers University of Technology. Available at: <http://dx.doi.org/10.1007/s11085-019-09935-9>
- [44] Reddy et al. (2021). Reevaluating the Cr Evaporation Characteristics of Ce/Co Coatings for Interconnect Applications. *ECS Transactions*, 103(1), 1899–1905. <https://doi.org/10.1149/10301.1899ecst>
- [45] Tomas, M. (2024). *Corrosion of Metallic Interconnects for Solid Oxide Fuel Cells: Mitigating Chromium Evaporation and Dual-Atmosphere Challenges*. Doctoral thesis, Chalmers University of Technology. ISBN 978-91-8103-038-9. https://research.chalmers.se/publication/540936/file/540936_Fulltext.pdf
- [46] Froitzheim, J., Ravash, H., Larsson, E., Johansson, L.-G. (2010). Investigation of Chromium Volatilization from FeCr Interconnects by a Denuder Technique. *Journal of The Electrochemical Society*, 157(9), B1295–B1300. <https://doi.org/10.1149/1.3462987>

-
- [47] Holcomb, G.R., Ziomek-Moroz, M., Cramer, S.D., Covino, B.S., Bullard, S.J. (2006). Dual environment effects on the oxidation of metallic interconnects. *Journal of Materials Engineering and Performance*, 15, 404–409. <http://dx.doi.org/10.1361/105994906X117198>
- [48] Gannon, P., Amendola, R. (2012). High-temperature, dual-atmosphere corrosion of solid-oxide fuel cell interconnects. *JOM*, 64, 1470–1476. <http://dx.doi.org/10.1007/s11837-012-0473-3>
- [49] Reddy, M. J. (2023). Metallic materials for solid oxide fuel cells and electrolyzers: Mitigating high temperature corrosion. *Doctoral thesis, Chalmers University of Technology*. ISBN 978-91-7905-832-6. <https://research.chalmers.se/en/publication/535305>
- [50] Egerton, R. F. (2005) The Scanning Electron Microscope, in *Physical Principles of Electron Microscopy: An Introduction to TEM, SEM, and AEM*. Boston: Springer, pp. 125–153. doi:10.1007/0-387-26016-1_5.
- [51] Zhou, W., Apkarian, R., Wang, Z. L. and Joy, D. (2007) Fundamentals of Scanning Electron Microscopy (SEM), in *Scanning Microscopy for Nanotechnology: Techniques and Applications*. New York: Springer, pp. 1–40. doi:10.1007/978-0-387-39620-0_1.
- [52] Goldstein, J. I., Newbury, D. E., Michael, J. R., Ritchie, N. W. M., Scott, J. H. J. and Joy, D. C. (2018) *Scanning Electron Microscopy and X-Ray Microanalysis*. New York: Springer.
- [53] Fournier-Salaün, M.-C. and Salaün, P. (2007) Quantitative determination of hexavalent chromium in aqueous solutions by UV-Vis spectrophotometer, *Central European Journal of Chemistry*, 5, pp. 1084–1093. doi:10.2478/s11532-007-0038-4.
- [54] H. Falk-Windisch, *Improved Oxidation Resistance and Reduced Cr Vaporization from Thin-Film Coated Solid Oxide Fuel Cell Interconnects*, PhD thesis, Chalmers University of Technology, Gothenburg, Sweden, 2017.
- [55] H. Kim, Fundamental Study on the Mechanism of High-Temperature Oxidation and Chromium Evaporation of Ferritic Stainless Steels for SOFC Interconnect Applications, PhD Thesis, Seoul National University, Seoul, South Korea, 2019.
- [56] J.G. Grolig, Coated Ferritic Stainless Steels as Interconnects in Solid Oxide Fuel Cells: Corrosion and Electrical Properties, Licentiate Thesis, Chalmers University of Technology, Gothenburg, Sweden, 2015.
- [57] P. Alnegren, M. Sattari, J.-E. Svensson, and J. Froitzheim, Temperature dependence of corrosion of ferritic stainless steel in dual atmosphere at 600–800 °C, *Journal of Power Sources*, vol. 392, pp. 129–138, 2018, doi: 10.1016/j.jpowsour.2018.04.088.

-
- [58] H. Falk Windisch, Improved Oxidation Resistance and Reduced Cr Vaporization from Thin-Film Coated Solid Oxide Fuel Cell Interconnects, Doctoral Thesis, Chalmers University of Technology, Gothenburg, Sweden, 2017.
- [59] S. Canovic, J. Froitzheim, A. Riess, M. Ranjbar, L.-G. Johansson, and J.-E. Svensson, Oxidation of Co- and Ce-Nanocoated FeCr Steels: A Microstructural Investigation, *Surface and Coatings Technology*, Vol. 215, pp. 62–74, 2013.
- [60] M. Stygar, P. Kurtyka, T. Brylewski, and W. Tejchman, Physicochemical and Mechanical Properties of Crofer 22 APU Ferritic Steel Applied in SOFC Interconnects, *Metallurgy and Foundry Engineering*, Vol. 39, No. 2, pp. 47–58, 2013.
- [61] M. Stygar, P. Kurtyka, T. Brylewski, and W. Tejchman, Physicochemical and Mechanical Properties of Crofer 22 APU Ferritic Steel Applied in SOFC Interconnects, *Metallurgy and Foundry Engineering*, Vol. 39, No. 2, pp. 47–58, 2013.
- [62] European Commission, Study on the Critical Raw Materials for the EU 2023 – Final Report, Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs, Publications Office of the European Union, Brussels, Belgium, 2023.

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