

CORROSION PROTECTION OF BIPOLAR PLATES IN CATION EXCHANGE MEMBRANE ELECTROCHEMICAL CELLS USING ADVANCED LASER CLAD MATERIALS

Doctoral Thesis – Summary

for the Scientific Title of Doctor

Politehnica University Timișoara

Field of Study: **Chemical Engineering**

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The doctoral thesis was developed based on research conducted within the Laboratory of Electrochemistry, Electrochemical Engineering, and Corrosion of the Faculty of Chemical Engineering, Biotechnology, and Environmental Protection (Politehnica University Timișoara – UPT). Additionally, the research activities were carried out at the Research Institute for Renewable Energies (UPT), at the Materials Science Laboratories of the Faculty of Mechanics (UPT) and Transilvania University of Brașov, as well as at the Department of Mechanical Engineering, Corrosion Laboratory of the University of Las Palmas de Gran Canaria, Spain, through an Erasmus+ mobility program.

The doctoral thesis entitled “Corrosion Protection of Bipolar Plates in Electrochemical Cells with Cation Exchange Membranes Using Advanced Materials Deposited by Laser Technology” is structured into nine chapters, each addressing a distinct topic. The chapters of the thesis are as follows: 1. Introduction; 2. Bipolar Plates – The Backbone of PEM Electrochemical Cells; 3. Laser Cladding; 4. Methods and Equipment; 5. Microstructural Characterization and Mechanical Properties; 6. Electrochemical Tests in Mildly Acidic Media; 7. Electrochemical Tests in Strong Acidic Media; 8. Interfacial Contact Resistance; 9. Conclusions and Personal Contributions.

Chapter 1, entitled “**Introduction**,” addresses general concepts regarding anthropogenic contributions that influence the global climate system, as well as the use of hydrogen as a potential paradigm shift in the supply of renewable energy.

Human activity has dramatically altered the environment, with the increase in greenhouse gas emissions being the main cause of global warming (Figure 1). Moreover, the energy sector is responsible for approximately three-quarters of greenhouse gas emissions and represents the key factor in mitigating the effects of climate change [1, 2].

In principle, using hydrogen as an energy source could address the issue of global warming. It is a flexible energy carrier applicable across all energy sectors that produces nearly zero emissions. In the long term, hydrogen has the potential to decarbonize highly polluting sectors, such as industry, transportation, and construction, and to serve as a means for long-term energy storage [3,4].

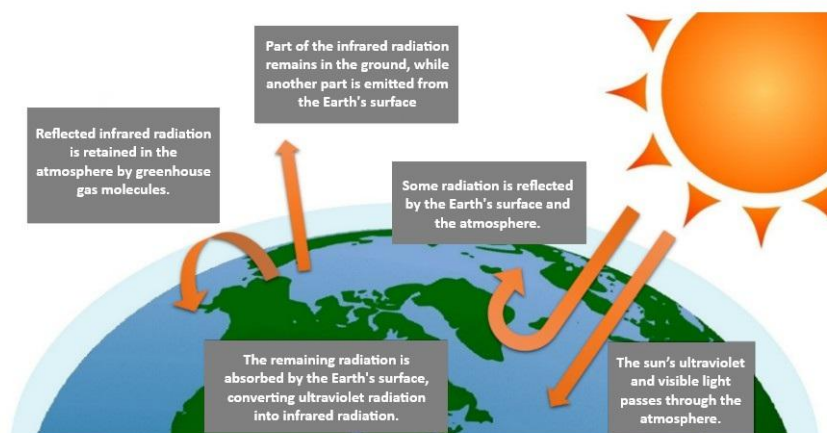


Figure 1: Natural Greenhouse Effect Explanation

A hydrogen economy based on renewable energy sources—including hydrogen production, storage, and conversion into electricity—is widely regarded as a promising solution that could, in principle, address the global greenhouse effect (schematically illustrated in Figure 2) [5], [6]. The strategy proposed by the International Energy Agency (IEA) for transitioning toward a near-zero-carbon energy economy includes the integration of effective action plans within the energy sector through the development of infrastructure, support for research and development projects, reduction of hydrogen production costs, implementation of new clean energy technologies, and improvement of existing ones [2], [4].

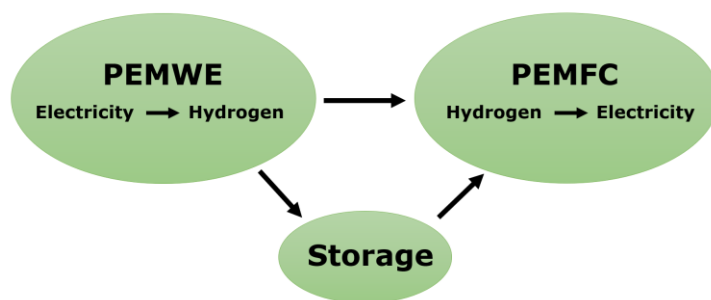


Figure 2: A simplified schematic representation of a hydrogen-based economy that utilizes hydrogen as an energy carrier.

Chapter 2, entitled “**Bipolar Plates – The Backbone of PEM Electrochemical Cells,**” presents the current state of knowledge in the field of bipolar plates, highlighting several theoretical aspects concerning modern manufacturing methods. It also provides a brief overview of PEM electrochemical cells, highlighting their main components and functions, as well as the reaction mechanisms occurring during operation.

Bipolar plates are essential to proton exchange membrane fuel cells (PEMFCs) and proton exchange membrane water electrolyzers (PEMWEs). In both electrochemical systems, the main functions of bipolar plates are to separate individual cells within the stack, conduct electric current

between adjacent cells, and remove heat from the reaction zones. Bipolar plates also play an essential role in distributing hydrogen and oxygen within PEMFCs and facilitating water distribution within PEMWEs [7].

Water electrolysis (schematically illustrated in Figure 3a) is an electrochemical process in which water is decomposed into hydrogen and oxygen through the application of an electric current, thereby converting electrical energy into chemical energy. At present, several technologies are employed for water electrolysis. Although their operating principles are similar, the main distinction lies in the type of membrane and electrolyte used. Based on the electrolyte employed, four well-known technologies are used for hydrogen production via electrolysis: alkaline water electrolysis (AWE), proton exchange membrane water electrolysis (PEMWE), anion exchange membrane water electrolysis (AEMWE), and solid oxide water electrolysis (SOWE). The hydrogen thus produced can subsequently be stored and transported for various industrial applications. The conversion of hydrogen back into electrical energy can be achieved either through its direct combustion in a thermal engine or by utilizing it in a fuel cell [8], [9].

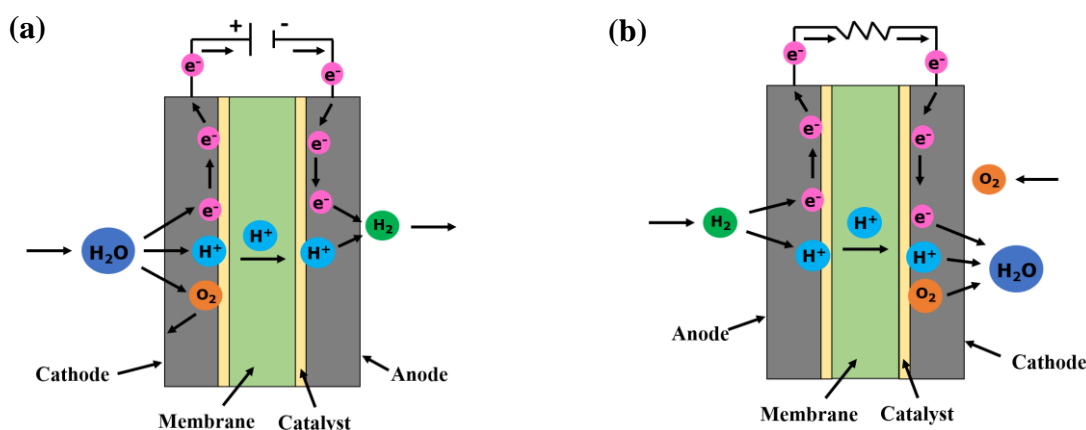


Figure 3. Schematic diagram of the operating principle of PEMWE (a) and PEMFC (b)

A fuel cell is an electrochemical „device” that converts chemical energy directly into electrical energy (along with a certain amount of heat) as long as the reactants—hydrogen (H_2) and oxygen (O_2)—are continuously supplied to the system. Consequently, fuel cells share similarities with both batteries, in that they rely on electrochemical reactions to generate energy, and internal combustion engines, which, unlike batteries, operate continuously by consuming a specific type of fuel. However, the analogy ends there. Unlike engines or batteries, a fuel cell does not require recharging, operates quietly and efficiently, and when hydrogen is used as fuel, it produces only electricity and water as by-products. For this reason, fuel cells have often been referred to as “zero-emission engines” [1].

Fuel cells represent a unique technology, providing an efficient, clean, and sustainable source of energy. Their operating principle is schematically illustrated in Figure 3b. Similar to electrolyzers, several fuel cell technologies have been developed. The fundamental operating principle remains the same, with the main distinction being the type of membrane and electrolyte employed.

Several well-established fuel cell technologies exist and are classified according to the type of electrolyte used. These include proton exchange membrane fuel cells (PEMFCs), alkaline fuel cells (AFCs), phosphoric acid fuel cells (PAFCs), molten carbonate fuel cells (MCFCs), and solid oxide fuel cells (SOFCs) [10].

The bipolar plates interconnect the cathode of one cell to the anode of the adjacent cell, creating a single-cell configuration. To achieve adequate output power, multiple cells are connected in series to form a stack. Consequently, bipolar plates account for approximately 75% of the stack's total volume and up to 45% of its overall cost [11]. Figure 4 illustrates the construction of PEM electrochemical cells.

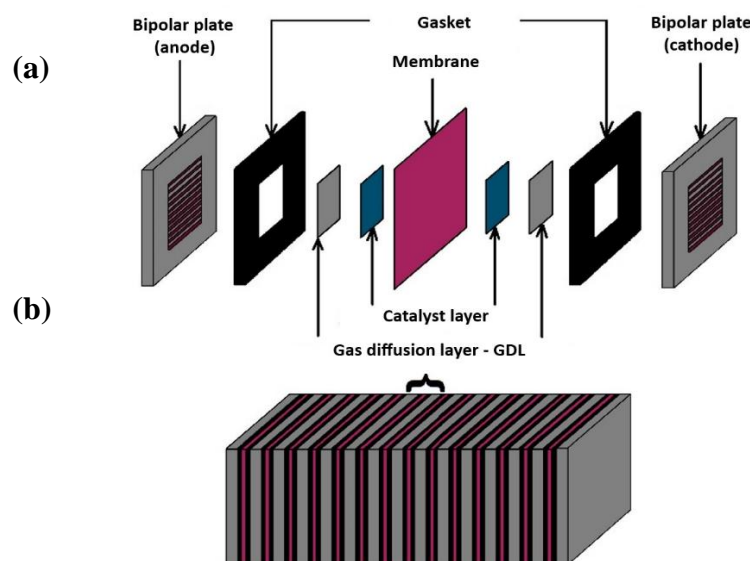


Figure 4: The components of PEM Electrochemical Cells (a) Single-Cell Configuration
(b) Complete Stack Assembly

The primary requirements for bipolar plates are thermal stability, mechanical strength, and corrosion resistance in the complex electrochemical environment of the cells. First and most importantly, they must possess adequate mechanical strength to withstand high operating pressures. They must also exhibit chemical stability and endure the alternating stresses resulting from the thermal expansion of the cell's components during operational cycles [12]. Additionally, they must demonstrate high corrosion resistance in the cell's working environment while avoiding the use of costly materials. To maintain low production costs, the selected material must be economically viable, and the manufacturing process must be suitable for large-scale production.

Materials used to develop bipolar plates must exhibit specific properties that can fulfill the aforementioned functional requirements simultaneously. A variety of materials have been used to fabricate bipolar plates, including graphite, uncoated or coated metals, and polymer composites with conductive fillers, such as metals or carbon [13]. So far, each material investigated has only demonstrated the ability to meet a subset of the specific requirements established by the U.S. Department of Energy (DOE). Therefore, exploring new materials for bipolar plates is essential to advancing PEM electrochemical cells with enhanced performance characteristics.

Various metals and metal alloys, such as stainless steel, aluminum, copper, titanium, nickel, and their alloys, have been investigated as potential candidates for the development of bipolar plates. However, due to the harsh operating conditions within PEM electrochemical cells, metallic bipolar plates exhibit several significant drawbacks, including surface corrosion and the dissolution of metal ions [6].

To overcome the issue of surface corrosion, various protective coatings have been investigated to enhance the corrosion resistance of metallic bipolar plates. In recent years, several coating fabrication techniques have been studied, including chemical vapor deposition (CVD), electrophoretic deposition, magnetron sputtering, cathodic arc deposition, electron beam evaporation, arc ion plating, and high-velocity oxy-fuel (HVOF) spraying, among others. Among the surface coating techniques for metallic substrates, laser cladding stands out as a modern and efficient method for producing coatings with improved surface properties. This technique offers the advantage of applying a particular material as a coating onto a selected substrate surface. Recent studies have demonstrated that laser cladding can produce high-quality coatings over large surface areas, with minimal heat-affected zones and significantly enhanced resistance to corrosion and wear [1].

A wide range of materials has been successfully deposited using various alloys (e.g., NiCrBSi, WC-Co/NiCrBSi, and Inconel 718) for different applications. NiCr-based alloys have received considerable attention due to their ability to produce high-quality coatings, excellent corrosion resistance, and cost-effectiveness [14, 15].

Chapter 3, entitled “**Laser Cladding**” addresses the current state of the art in the field of functional coatings, presenting theoretical aspects of various methods for producing metallic protective coatings. Among the different techniques some of them are discussed: chemical vapor deposition (CVD), physical vapor deposition (PVD), electroplating, thermal metallization, welding-based cladding, thermal spraying, and laser cladding. Laser cladding, the method employed in the experimental studies, involves generating a protective coating by melting a feedstock powder deposited onto the surface of a given substrate. The operating principle of this process is illustrated in Figure 5.

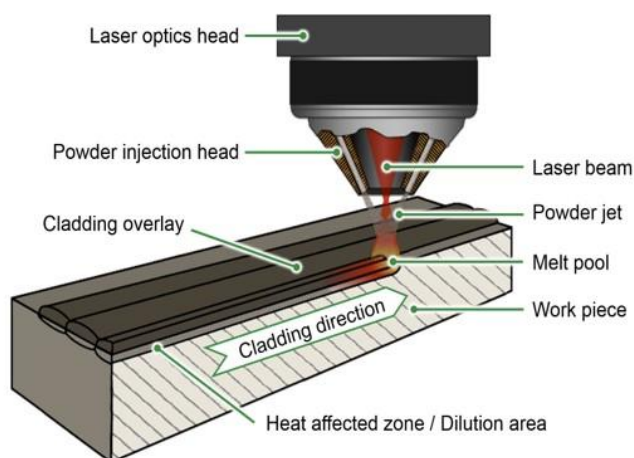


Figure 5. Deposition principle of laser cladding using powder as feedstock material

This technique generates a strong metallurgical bond between the deposited coating and the substrate. The feed material, in powder form, is melted and projected onto the substrate surface. The droplets flatten, overlap, and solidify rapidly upon impact, leading to the formation of a lamellar microstructure [16].

This chapter also discusses general considerations regarding surfaces preparation prior laser cladding, how deposition parameters influence the process, and materials reported in the literature for laser cladding applications.

Chapter 4, entitled "**Methods and Equipment**", describes the equipment and materials used to deposit NiCr(Ti) coatings with laser technology.

For the experimental studies, a COHERENT F1000 diode laser (Santa Clara, CA, USA) was employed with a maximum power of 1000 W and a wavelength of 975 nm. The powder was transported from the feeder through a hose to the PRECITEC WC 50 laser processing head (Gaggenau, Germany). An AT-1200 HPHV TERMACH feeding system (Haiger, Germany) facilitated powder delivery to the laser processing head. Argon was used as the carrier/protective gas. A six-axis CLOOS robotic arm deposited ten partially overlapping tracks onto the substrate. Figure 6 illustrates the equipment used for the manufacturing of coatings via laser cladding.

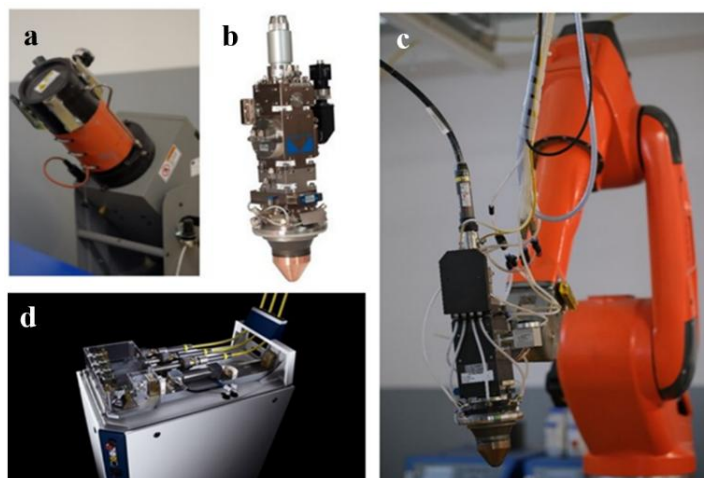


Figure 6. Laser cladding system: a) powder feeder; b) Precitec laser head; c) 6-axis robot; d) laser control unit

This chapter provides an overview of the methods used for the investigation and presents characterization techniques that provided essential information about the quality and performance of the materials used for developing bipolar plates. Several methods were employed to evaluate the performance of the NiCr(Ti) layers, including scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), X-ray diffraction (XRD), hardness measurements, and electrochemical methods in mild and strongly acidic media. Interfacial contact resistance (ICR) testing was also performed to evaluate the electrical conductivity of the deposited coatings.

Chapter 5, entitled "**Microstructural Characterization and Mechanical Properties**" describes the chemical composition and morphology of the feedstock powders and substrate material (low carbon steel). The chapter also provides a detailed description of the microstructural characterization of the coatings and the identification of phases within the laser clad coatings via X-ray diffraction (XRD). Additionally, it describes how the mechanical properties of the samples were determined through microhardness measurements.

From the cross-sectional SEM micrographs, it was observed that all samples exhibit a dense coating, free of cracks or other major defects. Three primary phases were identified: (i) the Ni-Cr matrix; (ii) a Nb- and Mo-rich phase, characteristic of Ni-Cr-based alloys; and (iii) a phase with a high Si content (for coatings without Ti addition) or a high Ti content (for coatings manufactured with Ti additions), corresponding to MC-type carbides.

The crystalline phases in the NiCr(Ti)-based coating were investigated by X-ray diffraction. For all samples, diffraction peaks corresponding to the NiCr- γ phase were observed. In the coatings with titanium additions, the presence of titanium was also confirmed alongside the NiCr- γ phase.

Microhardness measurements indicated that the increase in hardness values was attributed due to the formation of hard phases within the coatings. As the Ti content increased, so did the fraction of hard phases, which led directly to an increase in hardness.

Chapter 6, entitled "**Electrochemical Testing in Mildly Acidic Media**", presents the electrochemical evaluation of the coatings before and after chronoamperometric studies. The electrochemical protocol for all samples consisted of open-circuit potential (OCP) measurements, followed by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (Tafel) analysis. Subsequently, chronoamperometric tests were conducted in mildly acidic conditions under simulated anodic and cathodic environments of a PEM electrochemical cell, each for a duration of six hours. Afterward, EIS and Tafel measurements were repeated.

The electrochemical tests were carried out using a BioLogic SP-150 potentiostat/galvanostat (France). A conventional three-electrode electrochemical cell was employed, with the working electrode having an active surface area of 1 cm². The investigated materials served as the working electrodes, while platinum was used as the counter electrode. An Ag/AgCl (saturated KCl) electrode was used as the reference electrode. The electrolyte solution for the electrochemical tests consisted of 0.1 M Na₂SO₄ (acidulated with H₂SO₄ to pH 5), with 0.1 ppm F⁻, representing a mildly acidic environment. All samples were tested at 25 °C.

During PEM fuel cell operation, F⁻ ions are released into the system due to degradation of the proton exchange membrane. Accordingly, a concentration of 0.1 ppm F⁻ ions was added to the test solutions to simulate real operating conditions.

OCP, EIS, and Tafel measurements conducted prior to the chronoamperometric studies indicated that the addition of varying Ti additions (up to 10 %) to the base material led to an increase in corrosion resistance.

Chronoamperometric studies in mildly acidic media showed that the current densities decreased with increasing Ti content in the deposited coatings. The best performance was observed for the B + 10 % Ti sample, exhibiting a current density below 1 $\mu\text{A cm}^{-2}$ in both anodic and cathodic environments.

After exposure to chronoamperometric testing, it was observed that the corrosion resistance decreased proportionally by nearly one order of magnitude in both cathodic and anodic environments for all samples. Similar trends were observed in the Tafel parameters, with higher corrosion current densities measured after cathodic and anodic potentiostatic polarization.

Chapter 7, entitled “**Electrochemical Testing in Strongly Acidic Media**” presents the electrochemical evaluation of the coatings before and after chronoamperometric studies. The electrochemical protocol for all samples consisted of open-circuit potential (OCP) measurements, followed by electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (Tafel) analysis. Chronoamperometric tests were subsequently conducted in strongly acidic conditions under simulated anodic and cathodic environments of a PEM electrochemical cell, each for a duration of six hours. Following these tests, EIS and Tafel measurements were repeated.

The electrochemical tests were performed using a BioLogic SP-150 potentiostat / galvanostat (France). A conventional three-electrode electrochemical cell was employed, with the working electrode having an active surface area of 1 cm². The samples investigated served as the working electrodes, and platinum was used as the counter electrode. A saturated calomel electrode (SCE) was used as the reference electrode. The electrolyte solution consisted of 0.5 M H₂SO₄ with 2 ppm F⁻, representing a strongly acidic environment. All tests were conducted at 25 °C.

During PEM electrolyzes operation, F⁻ ions are released into the system due to degradation of the proton exchange membrane. Accordingly, a concentration of 2 ppm F⁻ ions was added to the test solutions.

OCP, EIS, and Tafel measurements conducted prior to the chronoamperometric studies indicated that the addition of varying Ti addition (up to 10 %) to the base material led to an increased corrosion resistance.

Chronoamperometric studies in strongly acidic media showed that current densities decreased with increasing Ti content in the coating layer. Under simulated cathodic conditions, the best performance was observed for the B + 3 % Ti, B + 5 % Ti, B + 7 % Ti, and B + 10 % Ti samples, each exhibiting a current density below 0.1 μA cm⁻². A different behavior was observed under simulated anodic conditions, where the current density increased by two orders of magnitude and varied significantly between the initial and final measurement points.

Following chronoamperometry, corrosion resistance increased by nearly one order of magnitude under cathodic conditions for all samples. Conversely, corrosion resistance decreased proportionally by several orders of magnitude under anodic conditions for all samples.

Chapter 8, entitled “**Interfacial Contact Resistance**” presents the studies conducted on the conductivity of NiCr(Ti)-based laser clad coatings. The compression force applied in this experiment ranged from 50 to 1500 N, corresponding to a compaction pressure of approximately 16 to 500 N cm⁻². Two configurations were considered in the experimental studies to measure interfacial contact resistances: one for the gas diffusion layer (GDL) alone and another for the combined sample + GDL.

The first configuration involved placing untreated carbon paper (GDL) between two Cu/Au plates. This assembly was then positioned between two insulating poly(methyl methacrylate) (PMMA) plates and placed in a hydraulic press. A direct current of 1 A was applied to the Cu/Au plates using a power supply, and the resulting voltage was measured with a precision multimeter. Figure 7(a) shows a schematic representation of the employed configuration.

For the second configuration, the carbon paper was placed in contact with the coated surface of the samples, which were then positioned between two Cu/Au plates. Then, the assembly was placed between two insulating PMMA plates and inserted into the hydraulic press. A direct current of 1 A was applied to the Cu/Au plates using a power supply, and the resulting voltage was measured with a precision multimeter. Figure 7(b) shows a schematic representation of the second configuration.

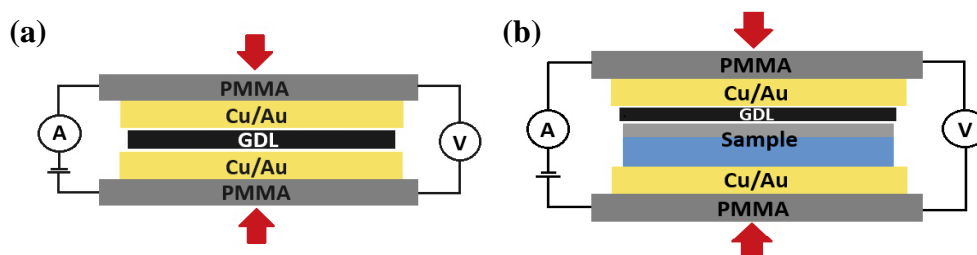


Figure 7. Schematic representation of the interfacial contact resistance process in the case of the gas diffusion layer (GDL) (a) and in the case of + GDL samples (b)

After conducting the interfacial contact resistance (ICR) tests, it was observed that the ICR value is primarily influenced by the surface conductivity of the sample at a given pressure. This conductivity is affected by the nature of the surface materials. Surface composition was the predominant factor at higher compaction forces. The experimental data indicated that the ICR increases with higher Ti additions to the base material due to the formation of a passive oxide film on the surface of coatings. The lowest ICR values in the tested sample were obtained for the NiCr-based samples containing 1.5% and 3% Ti at a compaction pressure of 140 N cm^{-2} .

Chapter 9 presents the general conclusions of this study and highlights the original contributions made through this work.

Based on the research conducted, the following conclusions can be drawn:

- New NiCr(Ti)-based protective coatings were successfully manufactured using a continuous laser cladding process, with potential applicability for bipolar plates in cation-exchange membrane electrochemical cells;
- Cross-sectional SEM micrographs revealed that all samples exhibit a dense coating, free of cracks or other major defects;
- Microstructural analysis identified three primary phases: (i) the Ni-Cr matrix; (ii) a Nb- and Mo-rich phase; and (iii) a phase with high Si content (for coatings without Ti) or high Ti content (for coatings produced with Ti additions), corresponding to MC-type carbides;
- X-ray diffraction (XRD) analysis identified the phases in the NiCr(Ti)-based coatings as NiCr- γ , and the presence of elemental titanium was confirmed, indicating that it was not fully transformed in the formation of secondary phases;
- An increase in Ti content within the coatings resulted in a higher proportion of hard phases and, consequently, an increase in hardness values;
- Electrochemical evaluation of the NiCr(Ti)-based coatings, both in mildly acidic and strongly acidic media (OCP, EIS, Tafel prior to chronoamperometry), showed that the

addition of varying Ti additions (up to 10 %) to the base material led to enhanced corrosion resistance compared to the base material;

- Chronoamperometric studies, in both mildly and strongly acidic environments, demonstrated that current densities decreased with increasing Ti content in the coatings;
- Lower performance was observed in both mildly and strongly acidic media upon retesting the samples after the chronoamperometric studies;
- Interfacial contact resistance was found to increase with higher Ti additions in the base material due to the formation of a passive oxide layer on the surface of the coatings. Among the tested samples, the lowest ICR values at a compaction pressure of 140 N cm^{-2} were achieved for NiCr-based coatings containing 1.5 % and 3 % Ti.

Individual contributions:

Considering the thesis's objectives and results, the personal contributions can be highlighted as follows:

- A literature study on the current state of laser cladding, including new processes and equipment, as well as a comparison of this method with other technologies for producing protective coatings. This study aimed to develop high-performance materials for PEM applications;
- The selection and study of NiCr-based materials and their applications in manufacturing protective coatings via laser cladding;
- The proposal to add Ti to NiCr-based powders to enhance performance with the aim of applying these new materials in the PEM field;
- Preparation of powders for laser cladding: weighing and homogenization;
- Assistance during the deposition of coatings using laser technology at the Materials Science and Engineering laboratories of UNITBV, Brașov;
- Preparation of samples for metallographic analysis and corrosion testing through grinding and polishing;
- Examination of cross-sections of the coatings using a metallographic microscope;
- Preparation of solutions for electrochemical testing;
- Perform electrochemical tests at the University of Las Palmas, Gran Canaria;
- Interpretation of experimental results and data processing;
- Determination of the optimal Ti concentration in NiCr-based coatings to develop new laser-clad materials with enhanced mechanical properties and increased corrosion resistance in mildly and strongly acidic environments with potential applications in PEM systems;
- Writing the thesis and publishing scientific articles.

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