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STUDIES AND RESEARCH ON THE CORROSION BEHAVIOR IN K_2SO_4 OF SOME LASER DEPOSITED COATINGS

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ABSTRACT

The paper presents a series of experimental investigations regarding the corrosion behavior in a K_2SO_4 – 0.5M solution of laser-deposited layers using a Ni-Cr-B-Fe-Al alloy, compared to the substrate material made of 1C45 steel. Corrosion resistance can be influenced by the deposition parameters, which determine the stress state, chemical inhomogeneity, defects, and discontinuities within the surface layer, as well as subsequent processing.

KEYWORDS: injected powder laser cladding, continuous wave laser, corrosion resistance

1. Introduction

Concerns regarding increasing the durability of carbon and alloy steel components, while also reducing energy consumption, labour, and materials, have led to the development of unconventional technologies for the surface treatment of active zones. These involve the use of concentrated energy beams (laser radiation, electrons, plasma).

The use of lasers in surface treatments offers a series of advantages [1]:

- It allows for the precise localization of the areas to be processed, regardless of their configuration, and the exact focusing of laser radiation through beam delivery and component positioning systems.
- It enables precisely controllable processing within very short time intervals, with minimal heat-affected zones (adjacent to the processing zone).
- It ensures the reduction of deformations, internal stresses, oxidation, and decarburization of components.
- It allows processing to be carried out within electrical or magnetic fields, or vacuum chambers.
- It provides a wide spectrum of properties to the processed surfaces by increasing hardness, wear resistance, fatigue resistance, and corrosion resistance.
- It enables full automation of the workflow.

In laser cladding, the interaction of the laser radiation with the workpiece can be achieved in the presence of a filler material that is either injected or pre-deposited in the laser-material interaction zone.

Evaluating the possibilities of laser cladding requires characterizing the involved elements: the processed material, the laser beam, and the working environment.

Thus, it is important to know the physical-mechanical characteristics of the processed material, along with the shape, dimensions, and precision required for the process.

The laser generator and the beam power density are selected depending on the absorption rate of the material and the transformations that occur.

The effects of the laser beam-material interaction depend on the actual distribution of energy across the processed surface.

Experimental determination of the laser beam dimensions and the spatial distribution of power density constitute a necessary condition for evaluating results and controlling the process.

Qualitative indicators of the laser-processed zone during the cladding process - such as the height of the deposited layer, its hardness, wear resistance, and corrosion resistance - are influenced by the processing parameters, namely power density, feed rate, powder feed rate, and laser action duration.

Thus, small changes in laser radiation absorption can lead to significant changes in the size of the molten pool formed on the processed surface.

Variations in the filler material feed rate can cause fluctuations in the geometry and microstructure of the deposited layer.

Consequently, by varying the processing parameters, the complex physical-mechanical characteristics of the deposited layers can be controlled.

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Laser cladding can be used in industry for hardening turbine blades in aircraft engines, valve seats in automotive engines, and lathe tools.

Through multi-layer deposition, the reconstruction of worn parts such as turbines, aircraft turbine shafts, and dies in the automotive industry can be achieved, thereby reducing material consumption, processing time, and the cost of subsequent machining [2-4].

Laser cladding with nickel-based alloys is utilized for components operating under high mechanical and thermal loads in aggressive environments.

They are resistant to atmospheric corrosion, water, alkaline solutions, salts, organic acids, and dry gases containing hydrochloric acid, hydrofluoric acid, and chlorine up to approximately 500 °C.

The aim of this paper is to characterize the corrosion behavior, in a K_2SO_4 – 0.5M, solution, of some laser-deposited layers using a Ni-Cr-B-Fe-Al, alloy, compared to the substrate material made of 1C45 steel.

2. Experimental conditions

The powder used for laser cladding is spherical, obtained by gas atomization, and presents the following chemical composition 8.9 %Cr; 4.5 %Fe; 5.1 %B; 2.4 %Al; 0.6 %Cu; and the rest Ni. The powder particle size ranged between 0.045-0.1 mm, and the hardness was HV0.1 = 12046 MPa [5].

The base material used in the experimental research is 1C45 steel in a quenched and tempered state. This material is widely used across many industrial sectors.

The laser cladding was performed in a single stage, with the powder being introduced directly into the laser-material interaction zone.

The implemented method involved lateral powder feeding because it is suitable for components of any shape.

It was observed that when the direction of the powder flow matches the direction of the component's movement, higher deposition efficiency is recorded, the melting depth is easier to control, and the occurrence of pores is less likely.

For the experimental depositions, a continuous-wave gas active medium system was used, specifically a Laser GT 1400W model.

The operating parameters used for the laser cladding of the Ni-Cr-B-Fe-Al alloy are presented in Table 1.

Metallographic analysis of the used samples was carried out using a Neophot 2 microscope with computerized data acquisition.

Microhardness determination was performed using a PMT-3 microhardness tester with a 100 g load.

Table 1. *Operating parameters used for laser cladding*

Processing Parameters	Sample 1	Sample 2 – with substrate preheating to 60 °C
Filler material feed rate, [mg/s]	105	63.9
Number of overlapping passes	4	4
Laser radiation power, [W]	1150	1150
Laser beam scanning speed across the processed surface, [mm/s]	11	11
Laser beam diameter, [mm]	1.8	1.8
Feed step, [mm]	1.5	1.5
Layer thickness, [mm]	0.59	0.42
Vickers microhardness, [MPa]	12500	6500

EDX analyses performed on the corroded samples were carried out using a Quanta 200 electronic microscope with a resolution of 30 nm, equipped with an AMETEK spectrometer.

Prior to the corrosion tests, the samples were mechanically ground and polished to limit contamination caused by previous processing. They were then connected to a copper wire and insulated with an epoxy resin.

Electrochemical studies were conducted using a PGP 201 Potentiostat/Galvanostat system, and data acquisition was carried out using VoltaMaster 4 software, version 7.10. The electrochemical cell used was a classic three-electrode setup: the working electrode (the studied material), the reference electrode (Hg/Hg_2SO_4 in a saturated K_2SO_4, solution, with a potential of +651 mV vs. NHE), to which all measured potentials were reported, and the auxiliary electrode (a pure platinum electrode). The active surface area of the samples was 1 $cm^2 \pm 0.3$ cm^2.

The electrolyte used (K_2SO_4 - 0.5 M) was prepared in the laboratory using analytical grade (p.a.) chemical reagents purchased from Sigma-Aldrich. The tests were conducted at room temperature 25 °C ± 1 °C. The electrochemical parameters of the studied electrolyte are shown in Table 2 below.

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Table 2. *Electrochemical parameters of the studied electrolyte*

pH	Electrical Conductivity [mS/cm]	Salinity [g/L]	TDS (Total Dissolved Salts) [ppt]
7.28	73.4	30.2	52.1

The applied electrochemical protocol was as follows:

- Evaluation of the free open circuit potential (OCP) for 3 hours, with a measuring period of 1.8 s.
- Potentiodynamic polarization (PD), general corrosion Rp and corrozion rate Vcorr; scan rate 1 mV/s; overvoltage 40 mV; OCP duration 1 min; determined measure 50 Rp; step duration 0.6 s; step amplitude 3 mV; total time 149 min; and initial scan cathodic.

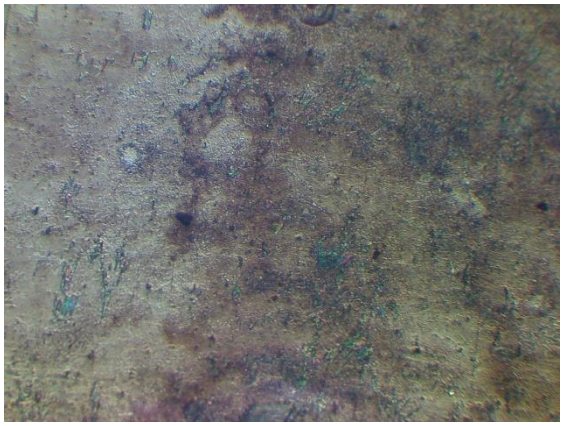
3. Results and discussions

3.1. Metallographic Analysis of Samples Tested for Corrosion in K_2SO_4 – 0.5M Solution

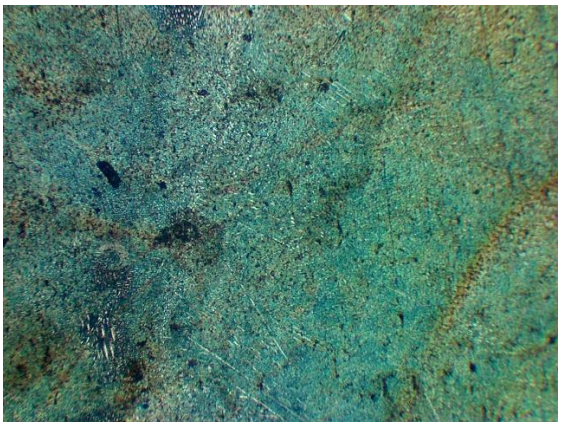
To highlight the corrosion behavior of the tested samples, a series of investigations were carried out using a Neophot 2 optical microscope, with computerized data acquisition, at a magnification power of 50x.

The obtained results are presented in Figure 1.

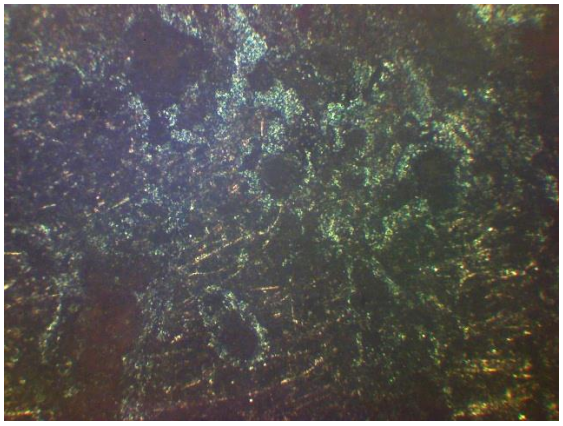
Figure 1.a, corresponding to sample P1, highlights localized corrosion in certain areas of the surface.



a) sample P1



b) sample P2



c) base material - sample P3

Fig. 1. *Metallographic analysis of the samples tested for corrosion in the K_2SO_4 – 0.5M solution, 50x*

Figure 1.b, corresponding to sample P2, which was prepared with substrate preheating, highlights reduced corrosion.

Figure 1.c, corresponding to sample P3 (the base material), highlights severe, uniform corrosion across the entire surface.

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Nevertheless, it can be observed that both samples produced by laser cladding with the Ni-Cr-B-Fe-Al alloy (P1 and P2) exhibit superior corrosion behavior compared to the 1C45 steel substrate.

3.2. EDX Microanalysis of Samples Tested for Corrosion in K_2SO_4 – 0.5M solution

EDX investigations carried out on the corroded samples were performed using a Quanta 200 electron microscope with a resolution of 30 nm, equipped with an AMETEK spectrometer.

Figure 2 shows the EDX microanalysis for sample P1 tested for corrosion in the in K_2SO_4 – 0.5M solution.

Analysing Figure 2 corresponding to sample P1, it can be observed that localized corrosion occurs in the form of pitting and spots. This typically manifests around defects such as pores, discontinuities, cracks, or inclusions.

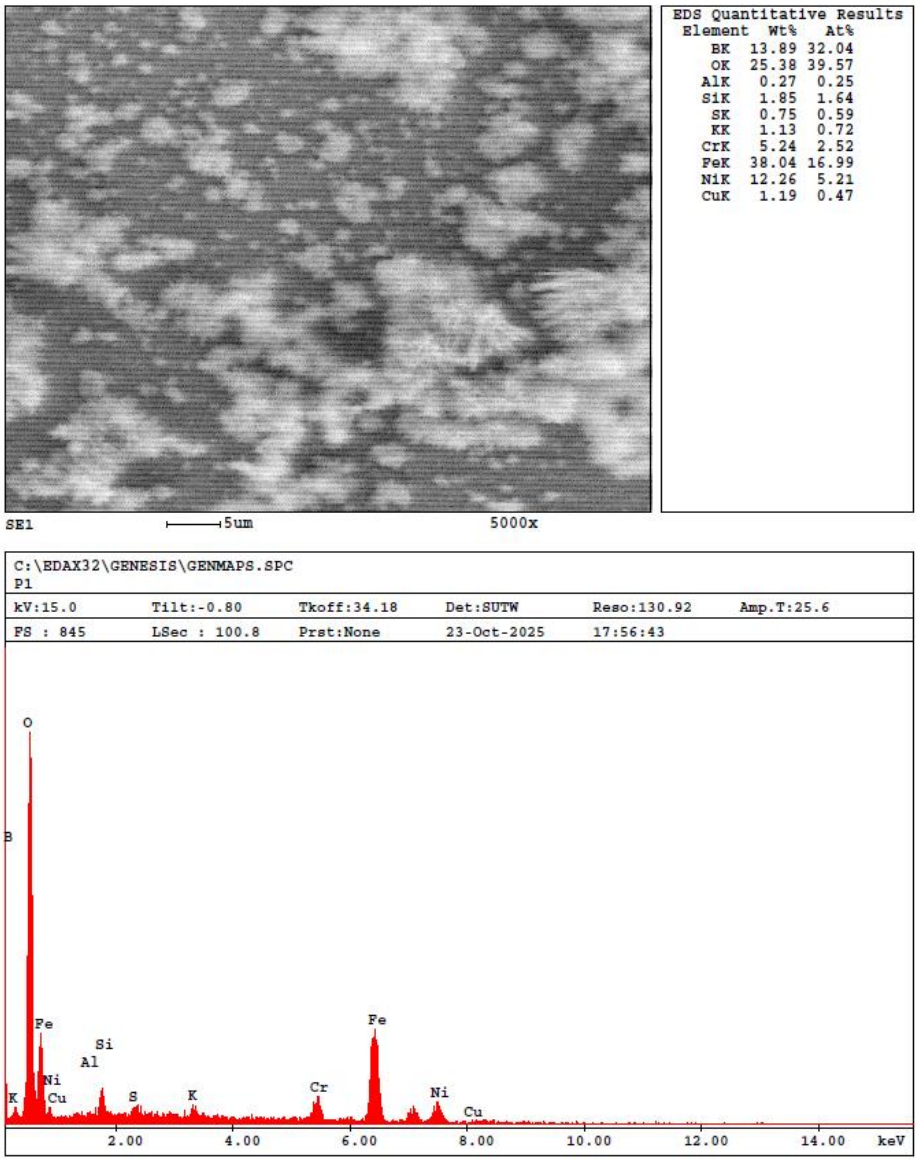

***Fig. 2.** EDX microanalysis for sample P1*

Figure 3 presents the EDX microanalysis for sample P2, prepared with substrate preheating at 60 ºC, tested for corrosion in the K_2SO_4 – 0.5M. A lower degree of corrosion can be observed compared to sample P1. This behavior is determined by the reduction in the cracking tendency of the deposited layer.

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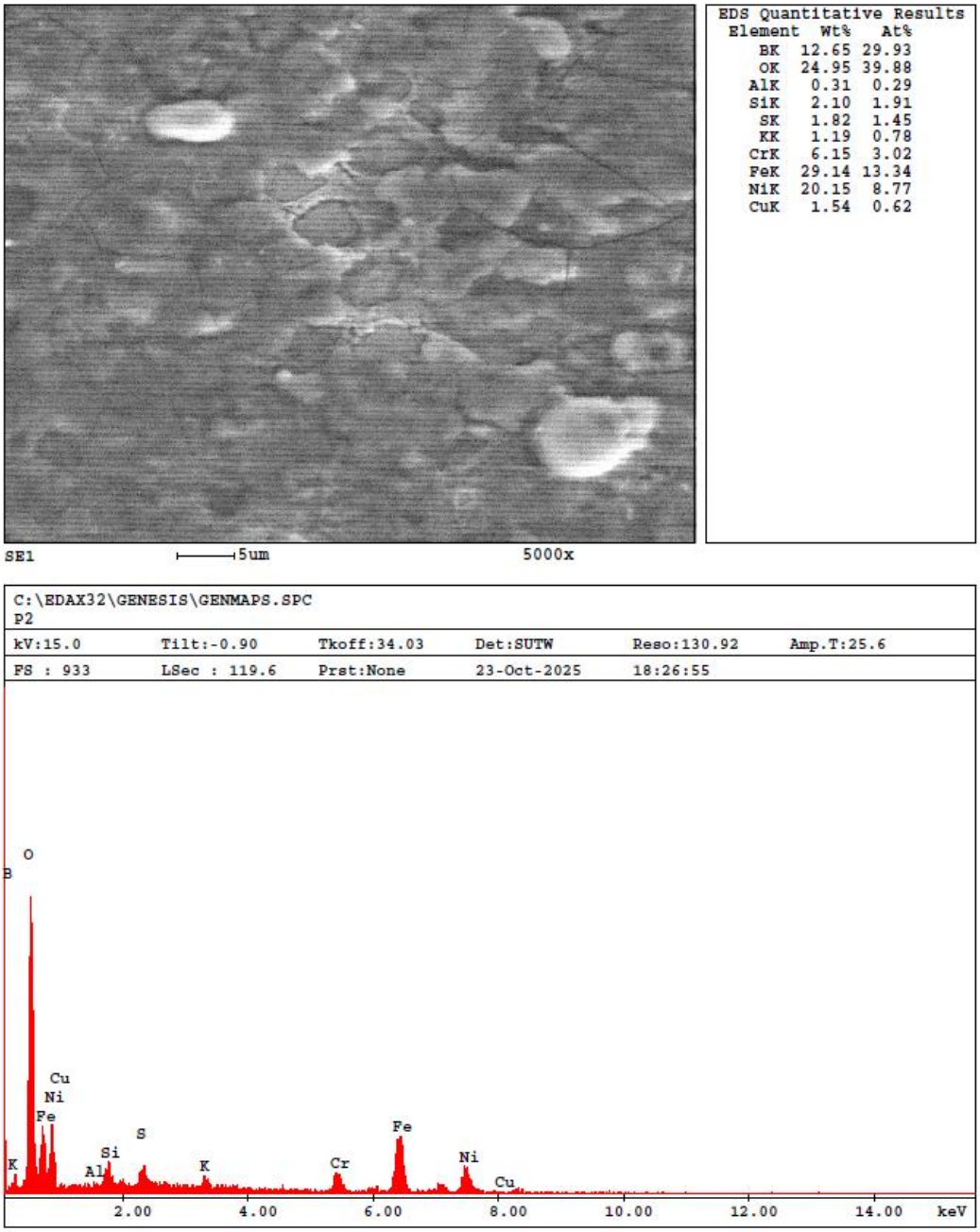

Fig. 3. *EDX microanalysis for sample P2, performed with substrate preheating*

Figure 4 corresponding to the EDX microanalysis for sample P3 (the base material), highlights generalized corrosion, characterized by the presence of corrosion products across the entire surface.

Analysing Figures. 2, 3, and 4, it can be concluded that the presence of chromium and aluminium in nickel-based alloys improves corrosion behavior. The formation of nickel, chromium, and aluminium oxides plays an important role in maintaining their passivity.

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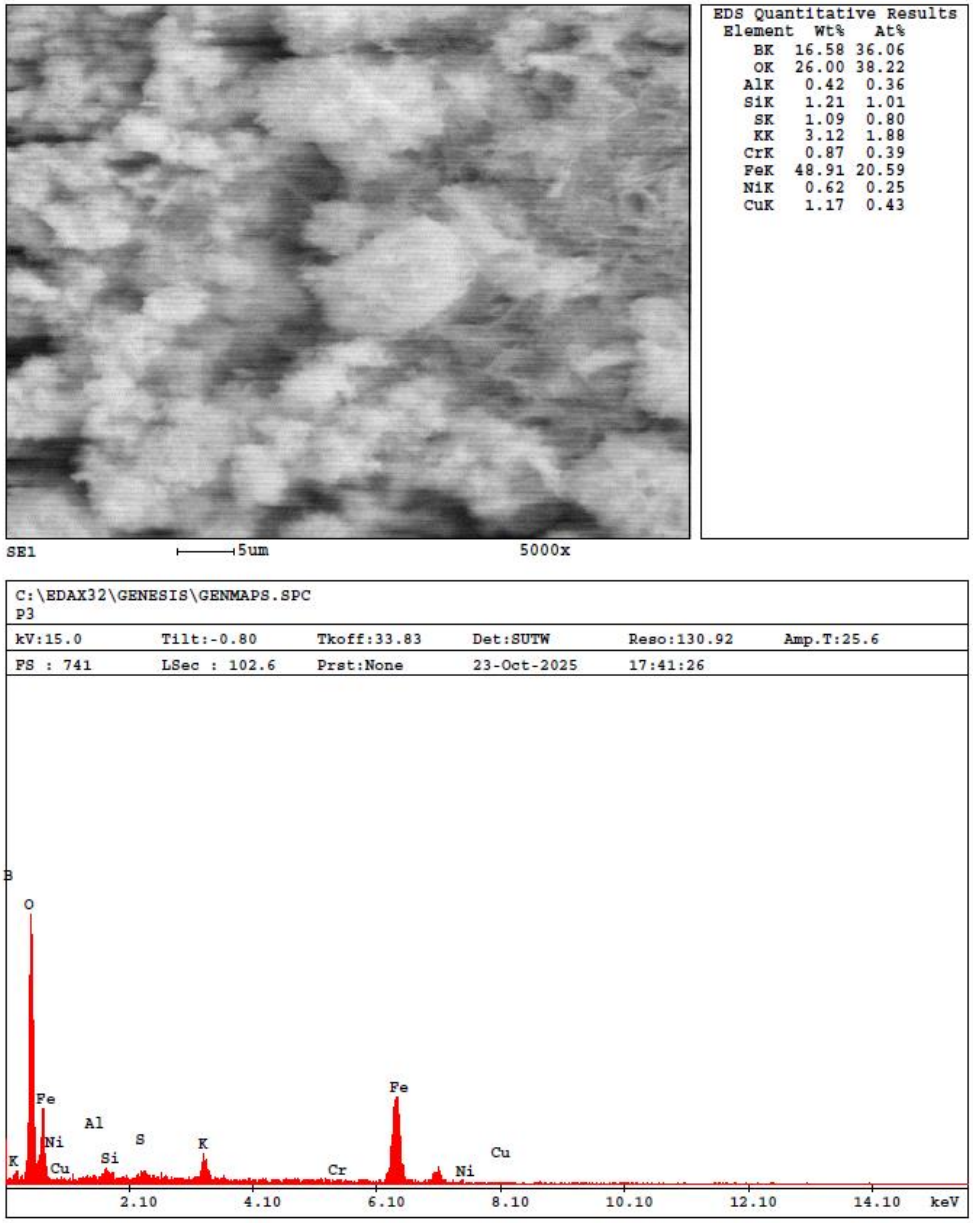

Fig. 4. *EDX microanalysis for sample P3, the base material*

Thus, the EDX investigations carried out on the corroded samples highlight the formation of corrosion products.

This aspect is confirmed by the appearance of oxygen in the resulting spectra. It can be observed that the oxygen percentage reaches its highest value in the base material, where corrosion was most intense.

Furthermore, the percentage of nickel, chromium, and aluminium decreases in sample P1 compared to sample P2 (prepared with substrate preheating), which confirms the appearance of oxides on their surfaces and a differing resistance to corrosion.

3.3. Evaluation of Free Potential (OCP) or Open Circuit Potential

Testing for each sample began with monitoring the free open circuit potential (OCP) after immersion in the K_2SO_4 – 0.5M, solution, aiming to observe their interaction with the corrosive solution. OCP monitoring was performed until a stationary value was reached.

Figure 5 shows the time evolution of the free OCP potential for the analysed samples.

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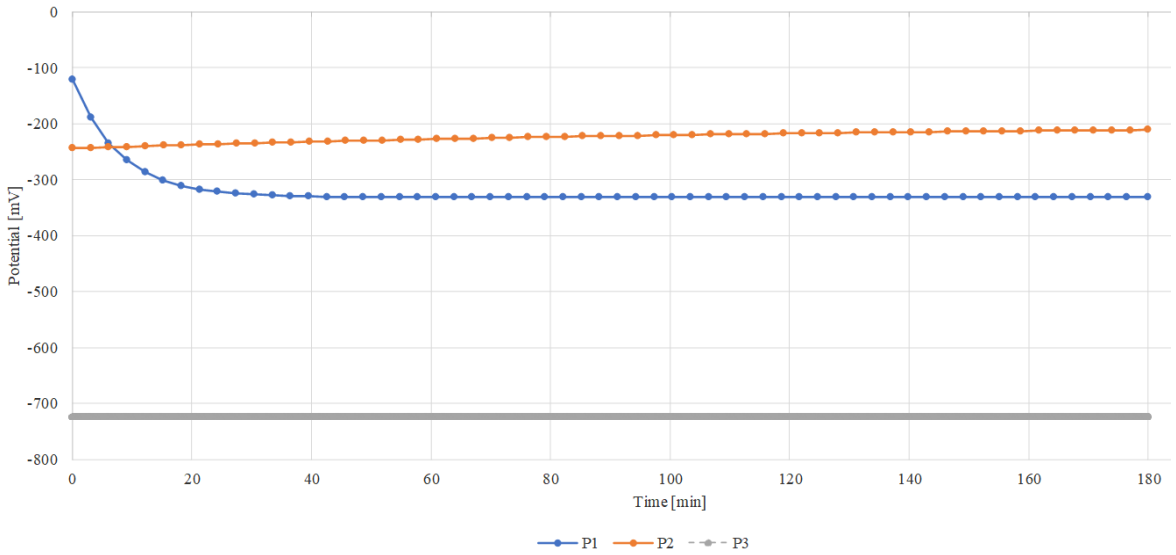

Fig. 5. *Time evolution of the free potential (OCP)*

Analysing Figure 5 regarding the time evolution of the free OCP potential, it can be observed that for sample P2, the corrosion potential stabilizes after approximately 150 minutes from immersion. Its more positive value, ranging between – 243.929 mV and – 210.913 mV indicates a greater tendency toward passivation, which reduces the corrosion process in the 0.5M K_2SO_4 solution, compared to the potential ranging between – 121.61 mV and – 331 mV for sample P1, and – 724 mV for sample P3.

3.4. Potentiodynamic method

This is used to study the kinetics of corrosion processes to determine the passivation range of a material after immersion in a corrosive electrolyte. From the analysis of the polarization curves, the mechanism and rate of the occurring corrosion reactions can be characterized [6, 7].

Figure 6 shows the evolution of polarization resistance over time.

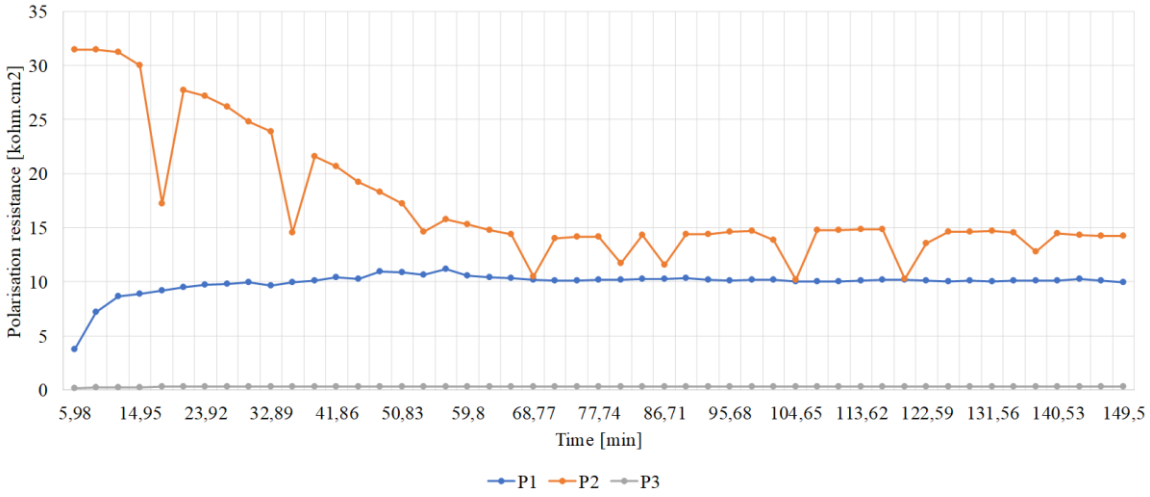

Fig. 6. *Evolution of polarization resistance over time*

Analysing Figure 6, it can be noted that for sample P1, the polarization resistance ranges between 3.77736÷9.9695 KΩcm^2, for sample P2 it varies between 31.4795÷14.2099 KΩcm^2, and for sample P3 it ranges between 0.136786÷0.312298 KΩcm^2. Thus, it can be concluded that sample P2, where the polarization resistance exhibits the highest values, presents the best corrosion resistance.

In the laser cladding process, depending on the parameters used, residual stresses can form between the substrate and the deposited material. These can influence the corrosion behavior. Compressive

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stresses appear within the deposited material, while tensile stresses form in the heat-affected zone.

These stresses are generated by the difference in the thermal expansion coefficient between the substrate and the deposition material, as well as by the temperature gradient that occurs during solidification. If the value of these stresses exceeds the ultimate strength of the material, it can lead to crack formation, thereby influencing corrosion behavior.

Thermal stresses can also lead to crack formation.

One way to avoid these shortcomings is to preheat the samples in a furnace, with a flame, or via induction. This leads to a reduction in the temperature gradient between the deposition material and the base material during cooling, and increases melting and deposition rates. Consequently, the productivity of the process is also improved. A larger dilution zone can occur in this case. This aspect can be limited by increasing the deposition speed.

Stresses can also be regulated by establishing and controlling the absorbed power, the track formation speed, and the feed rate.

Sample P2, produced with substrate preheating, is characterized by a lower stress state, which led to better corrosion behavior.

In the case of sample P1, where deposition was carried out without substrate preheating, the corrosive attack occurred along the cracks caused by the presence of residual stresses.

Nevertheless, it can be observed that both samples produced by laser cladding with the Ni-Cr-B-Fe-Al alloy (P1 and P2) exhibit superior corrosion behavior compared to the 1C45 steel substrate.

Figure 7 shows the evolution of the corrosion rate over time.

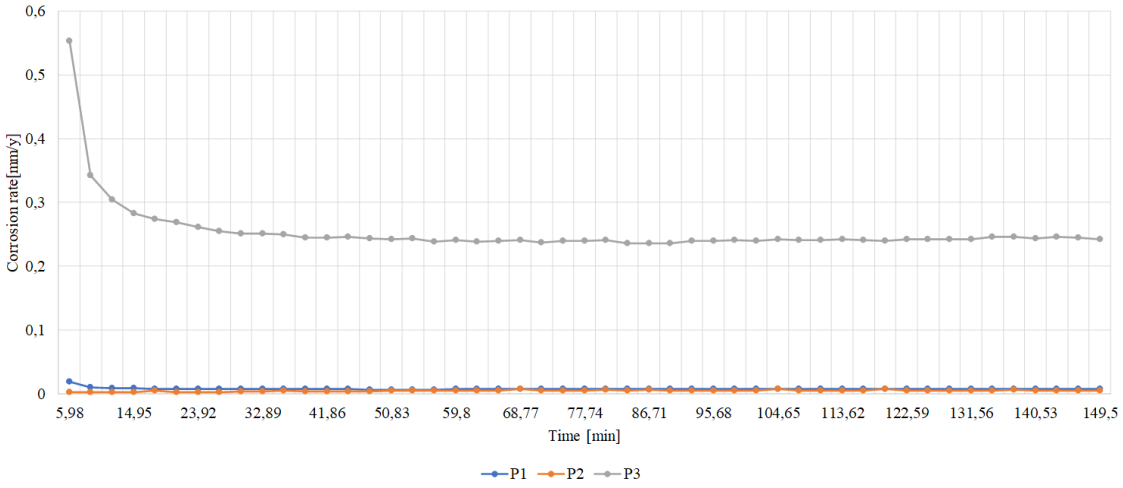

Fig. 7. *Evolution of corrosion rate over time*

Analysing Figure 7, it can be observed that the corrosion rate for sample P1 is between 0.018604÷0.007049 mm/year, for sample P2 it varies between 0.002232÷0.004945 mm/year, and for sample P3 it is between 0.552533÷0.242009 mm/year. Thus, sample P2 exhibits the best corrosion resistance in the 0.5M K_2SO_4.

The laser-deposited samples P1 and P2 exhibit a much lower corrosion rate compared to the substrate material.

Corrosion behavior can be influenced by the existence of pores and cracks within the deposited layer.

The presence of non-metallic inclusions, undispersed hard phase particles, and structural changes induced by laser cladding (imperfections such as dislocations or twins, which influence the stress state) can also affect corrosion behavior.

Table 3 presents the parameters of the solution after corrosion.

Table 3. *Electrochemical parameters of the electrolyte after corrosion*

Sample Code	pH	Electrical Conductivity [mS/cm]	Salinity [g/L]	TDS (Total Dissolved Salts) [ppt]
Initial	7.28	73.4	30.2	52.1
P1	8.02	75.4	21	53.6
P2	7.82	75.2	21.9	53.4
P3	7.75	75.7	19.9	53.8

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Analysing Table 3, an increase in the pH of the solution after corrosion can be observed. An increase in TDS can also be noticed as a result of the dissolution of the surfaces subjected to corrosion in the aggressive environment. Sparingly soluble compounds may form, which, by accumulating on the surface of the samples, can influence corrosion behavior. Thus, if the deposited film is compact, the surface is protected; if it is porous and permeable, it becomes corroded. In the case of sample P2, the total dissolved salts have the lowest value. An increase in electrical conductivity and a decrease in solution salinity after corrosion are also recorded.

4. Conclusions

The research conducted led to the formulation of the following conclusions:

- Laser cladding constitutes a more efficient alternative for modifying the properties of surface layers than conventional treatment methods (thermal spraying, welding, electrochemical deposition, electrical discharge in gases) because it ensures precisely controllable processing of parts with any configuration, under conditions of low filler material consumption, while achieving a strong metallurgical bond with the substrate, without deformations, and with reduced subsequent machining expenses.

- By controlling the processing parameters, the required physical and mechanical properties for service conditions can be imparted to the surface layer of components; the thickness of the laser-deposited layer must be correlated with the stresses arising from operation under specific known or foreseeable conditions; complex interactions may occur under static or cyclic loading, in corrosive environments, or at elevated temperatures.

- When the feed rate of the filler material is increased to 105mg/s for sample P1, the energy available for melting the substrate decreases, leading to an increase in the thickness and hardness of the deposited layer, as well as a reduction in dilution; residual stresses develop, resulting in the formation of cracks in the deposited layer, which in turn leads to a decrease in corrosion resistance.

- Preheating the substrate to 60 °C in the case of sample P2 and reducing the powder feed rate to 63.9 mg/s results in a reduction of the deposited layer thickness, a decrease in hardness, and a reduction in residual stresses, which leads to better corrosion behavior in the 0.5M K_2SO_4 solution. The dilution zone is larger in this case.

- Since the laser beam provides the energy required to melt the filler material, it is useful to correlate the scanning speed with the filler material feed rate; this, the higher the feed rate, the more the scanning speed should be reduced.

- Laser-deposited samples P1 and P2 with the Ni – Cr – B – Fe – Al alloy exhibit a much better corrosion behavior compared to the substrate material made of 1C45 steel.

- With an increase in the concentration of chromium, nickel, and silicon within the deposited layer, corrosion resistance improves.

- Corrosion can be exacerbated by the lack of chemical or physical homogeneity on the surface, by defects caused by the deposition parameters (pores, discontinuities, cracks), or by subsequent processing methods (which influence roughness and the stress state).

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