

Article

Evaluation of the Wear Properties and Corrosion Resistance of 52100 Steel Coated with Ni/CrC by Cold Spraying

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Abstract: Coatings deposited by the cold particle gas spray method have shown significant potential for enhancing the properties of metals. We investigated the wear characteristics and corrosion behavior of 52100 steel coated with a mixture of Ni/chromium carbide (Ni/CrC) particles. These coatings exhibited high density and were devoid of cracks, closely adhering to the substrate’s surface. Microscratch resistance testing revealed strong resilience, with the apparent friction coefficient exhibiting multiple peaks as the displacement varied. The determination of the friction coefficient, utilizing linear and rotational sliding tests, displayed a brief transition period. This occurred as the apparent contact area expanded until it reached an equilibrium state, with the large asperities being smoothed out and the remaining particles on the sliding track acting as an abrasive material, resulting in higher friction coefficient values. Electro-corrosion tests confirmed the near-intact condition of the deposited layer. Few compounds were detected in the electrolyte solution, resulting in significantly lower oxidation in the layer compared to the base material.

Keywords: coatings; cold spray; microstructural analysis; wear analysis; corrosion resistance



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1. Introduction

Surface coatings are used to enhance the wear, corrosion, and high temperature resistance of materials subjected to harsh working conditions. This study explores the impact of using the cold spray method to improve the wear and corrosion characteristics of coated 52100 steel [1–3]. The cold spraying method (CS) is attractive due to the possibility of producing dense coatings with low porosity at very low temperatures. This is only possible through this method, so that even at temperatures of approximately 700 °C, the deposited layer adheres excellently to the surface of the base material while preserving the properties of the powders used as raw materials. Due to these advantages and the wide range of powders with various properties that can be found at established companies in this field, the cold deposition method is excellent both for improving the properties of the materials and for the reconditioning processes of the various components [4–6]. This technique processes particles in a solid state and utilizes high kinetic energies generated by accelerating the particles in supersonic gas to deposit particles. The particle temperatures remain

below their melting point, making CS a unique solid-state deposition process [7]. Upon impact, the particles undergo plastic deformation and adhere to the substrate through metallurgical bonding and/or interlocking [8]. Improvements in deposition technology and powders thus cater to a wide range of desired coating properties for diverse needs and applications [9]. In this work, we aim to contribute to the field of protective coatings by investigating the potential of the CS deposition method to improve the wear and corrosion resistance of 52100 steel. We assessed the corrosion resistance and wear properties of a Ni/CrC blend coating deposited by CS on 52100 stainless steel and performed microstructural analyses to investigate the mechanisms involved in these processes.

The specialized literature provides valuable studies that confirm the fact that the cold spray method produces layers that adhere very well to the surface of the base material and increase its properties. For example, in the research carried out by Maier et al. [10], chromium-based coatings that reached a layer thickness of approximately 51 μm were deposited through CS. The hardness of the deposited layer was found to be higher than that of the powder particles due to the plastic deformation that occurred during deposition. These deposits also showed very good resistance to oxidation up to 1300 $^{\circ}\text{C}$. The XRD analysis showed that the early stages of the oxidation of Cr occurred at 1000 $^{\circ}\text{C}$ after 7 min of exposure, and at 1200 $^{\circ}\text{C}$, oxidation became more aggressive showing Cr_2O_3 peaks with the minimum intensity of the BCC-Cr peaks. Goanta et al. [11] used XRD to investigate the chemistry of AISI 52100 alloy steel coated with a chromium and nickel carbide mixture by cold spraying. They found the predominant and secondary phases in the coating to be NiCr and Cr_3C_2 , respectively. In the base material, the predominant phase was Fe, and the secondary phases were composed of Fe and Cr_3C_2 . The alloying elements that make up this type of steel give it superior properties and, at the same time, allow the creation of coatings to increase its properties even more, as can be seen in the research carried out by Fernandes et al. [12], where wear and corrosion tests for AISI 52100 alloy steel coated with niobium carbide were carried out. A (very high) hardness value of approximately 27 GPa and elastic modulus of 361 GPa were reported. These values were confirmed in other studies [13]. The NbC coating improved the corrosion behavior in 3.5% NaCl solution that involved potentiodynamic polarization tests with applied potentials up to 250 mV. Significant corrosion damage was observed at higher potentials where the electrolyte penetrated through the layer and reached the substrate. Rahbar et al. [14] conducted research on the wear resistance of 52100 steel sprayed with chromium by friction stir welding (FSW) and found the hardness to be approximately 791 HV. They found the wear resistance of the samples to be seven times higher, with a wear rate of 0.02 mg/m. The coating had low porosity and a uniform and refined microstructure with a layer thickness of 200 μm after FSW (the initial layer thickness was 300 μm , after the spraying). Romero et al. [15] deposited diamond-like hydrogenated carbon (aC:H) coatings on AISI 52100 steel, resulting in deposits with a thickness of 3 μm with a hardness of up to 19 GPa, showing good adhesion properties reaching a critical load of 72.6 N. Comparative studies of oxidation behavior were carried out by Karaoglanli [16] who deposited approximately 100 μm CoNiCrAlY layer thickness on Inconel-718 through cold spraying (CS), atmospheric plasma spraying (APS), detonation spraying (D-gun), and high-velocity oxygen fuel (HVOF). The highest resistance was achieved with the CS technique which, according to SEM images, showed a much lower oxide content and porosity in the structure. After 100 h, CS showed a thermally grown oxide (TGO) depth of approximately 6 μm , immediately followed by HVOF with almost 7 μm , and at the opposite end of performance showed D-Gun with a TGO thickness of about 12 μm . The deposition temperature plays an extremely important role in the creation of layers that adhere very well to the surface of the base material and, at the same time, preserve the properties of the powders used. Various studies [17] emphasize deposition parameters; for example, the influence of deposition parameters on tribological behavior was studied by Pukasiewicz et al. [18] for cold-sprayed FeMnCrSi alloy coatings. The coatings were carried out with N_2 using 600 $^{\circ}\text{C}$, 900 $^{\circ}\text{C}$, 1000 $^{\circ}\text{C}$, and 1100 $^{\circ}\text{C}$ and with He using 600 $^{\circ}\text{C}$ as the gas processing

temperature. Spraying with N₂ at 1000 °C resulted in the best wear performance, with WC and Al₂O₃ spheres. The microstructural properties showed compressive residual stresses of approximately 460 MPa with lower deposition efficiency in the case of deposition at a lower temperature of N₂.

With the purpose of enhancing the properties of the base material in terms of layer adhesion, microstructure, hardness, and corrosion resistance, the purpose of this study is to highlight the properties of Cr/C-based powders that have been deposited using the cold spraying method. This is in order to provide a more comprehensive picture of the performances that have been obtained by making these deposits.

2. Materials and Methods

2.1. Coating and Parameters

The coatings consisted of a mixture of Ni and CrC particles. They were deposited using a VRC, Gen-III CS machine (VRC Metal Systems, 600 N Ellsworth Rd, Box Elder, SD 57719, USA). The powders used for these deposits are commercially available and can be found under the name WIP-C1. Table 1 shows the chemical composition of WIP-C1 powders and Table 2 shows their mechanical properties [19–21]. A bond coat using WIP-BC1 powders was deposited before applying the WIP-C1 coating. The spray conditions are presented in Table 3; they can also be found in the references [11,22,23]. The machine-finished surface was abraded by hand with ScotchBrite™ and wiped with ethanol. The bond layer (WIP-BC1) was sprayed onto the surface with the nozzle oriented at 60° to the surface.

Table 1. Chemical composition of WIP-C1 powders [18–20].

Composition (wt%)	Co (%)	Cr (%)	Fe (%)	Ni (%)	W (%)	Al (%)	B (%)	Mo (%)	Si (%)	V (%)	C (%)
WIP-C1	0.02	68.45	0.08	27.84	0.01	0.06	0.04	0.01	0.43	0.01	3.00

Table 2. Mechanical properties of WIP-C1 powders [18–20].

Material	Geometry	Hardness	Porosity
Carbide	Spherical	375–425 [HV]	<1% (N ₂)

Table 3. Deposition parameters for cold-sprayed coatings [11].

Parameter	Value
Gas	Nitrogen
Pressure	6.2 MPa (900 psi)
Temperature	675 °C
Nozzle ID	WC NNZL0060
Nozzle throat size	2 mm
Powder feeder speed	8 rpm
Powder feeder gas flow	105 slm
Standoff distance	25 mm
Spray angle	90°
Nozzle traverse speed	250 mm/s
Nozzle step distance	0.5 mm
Layer thickness	0.127 mm
Target coating thickness	0.508 mm
Powder	WIP-C1
Bond coat	WIP-BC1 and 60°

2.2. Microstructural Analysis

In order to study the morphology of the surfaces, a Quanta 200 3D 20 kV dual beam (FEI, Brno, Czech Republic) scanning electron microscope (SEM) with a large field detector (LFD) spot size of 5 mm and a working distance of up to 15 mm was used. Metallographic samples were cut to standard sizes according to wear, microindentation, and scratch and corrosion tests. To perform surface and cross-section morphological analyses, rectangular samples were embedded in resin round specimens, and after which grinding was performed at different grain sizes, up to a grain size of 2500P, and metallographic etching was performed with Nital reagent with a chemical concentration of 10% HNO_3 .

2.3. Wear/Mechanical Analysis

Microindentation and microscratch and friction testing were carried out using the CETR UMT-2 tribometer (Bruker, Billerica, MA, USA) in its various appropriate modes. Microindentation was used to determine the hardness of the coated and the uncoated samples according to the ASTM E18 standard [24]. The microscratch resistance of the coatings was determined according to the ASTM D7187 standard [25]. Friction tests were carried out both in linear and rotational testing modes according to the ASTM 132 standard [26]. The dimensions of the samples used were between $\varnothing 64 \times 10$ mm for the determination by rotation movement and $30 \text{ mm} \times 30 \text{ mm} \times 10 \text{ mm}$ for the determination by translation motion; they were $20 \text{ mm} \times 20 \text{ mm} \times 10 \text{ mm}$ for the microscratch and indentation tests. In Figure 1, we can see a sample mounted on the table of a CETR UMT-2 tribometer.

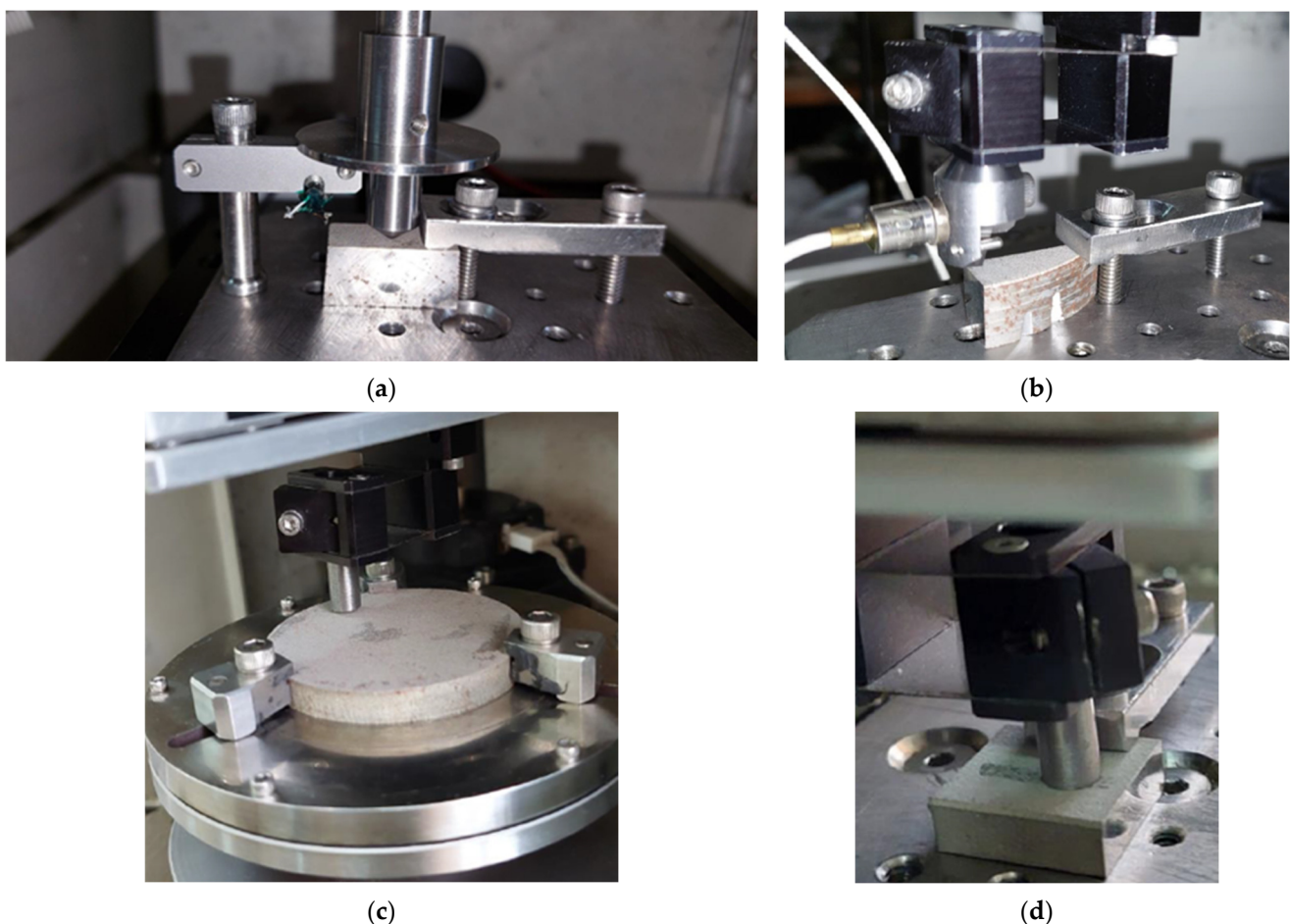


Figure 1. Tested samples on the CETR UMT-2 tribometer: (a) microindentation; (b) microscratch test; and (c,d) friction test.

The substrate used in this work was AISI 52100 steel, which is alloyed with chromium. Due to its high carbon content, it has high hardness, and thus it is characterized as a high-wear-resistance material. Table 4 shows the chemical composition of AISI 52100 alloy steel and its equivalents [27–29].

Table 4. Chemical composition of AISI 52100 alloy steel and its equivalents [22,23,27].

Standard	Type	Fe (%)	C (%)	Mn (%)	P (%)	S (%)	Si (%)	Ni (%)	Cr (%)	Cu (%)
AISI	52100	bal.	0.940	0.340	0.009	0.007	0.200		1.400	
AISI [27]	316L	bal.	0.022	1.583	0.037	0.0004	0.516	12.710	17.560	
ASTM A295 [30]	52100	bal.	0.93–1.05	0.25–0.45	0.025	0.015	0.15–0.35	0.25	1.35–1.60	0.30
DIN 17230 [31]	100Cr6/ 1.3505	bal.	0.90–1.05	0.25–0.45	0.030	0.025	0.15–0.35	0.30	1.35–1.65	0.30
IT G4805 [32]	SUJ2	bal.	0.95–1.10	0.50	0.025	0.025	0.15–0.35		1.30–1.60	
BS 970 [33]	535A99/ EN 31	bal.	0.95–0.10	0.40–0.70			0.10–0.35		1.20–1.60	

2.4. Corrosion Analysis

The corrosion resistance was determined by the electrochemical tests. The electrolyte solution was obtained from distilled water with 0.5 mol/L H₂SO₄ and 0.5 mol/L HNO₃, pH = 3.4. All electrochemical experiments were carried out using an electrochemical workstation Parstat 4000 (Ametek, Sao Paulo, Brazil) potentiostat/galvanostat connected to the three-electrode setup composed of the working electrode—the test sample—the reference electrode—the saturated calomel electrode (SCE)—and the counter electrode—the platinum electrode. The electrochemical test procedure started by measuring the open circuit potential (OCP) for 1 h after exposure of the working electrode to the solution at room temperature. Next, electrochemical impedance spectroscopy (EIS) tests were conducted in the frequency range of 1000–0.01 Hz. The results were fitted using ZSimpWin (version 3.22, Ametek, Sao Paulo, Brazil) software to obtain Nyquist and Bode plots. Potentiodynamic polarization tests were performed from −0.3 V vs. open circuit potential (OCP) to +0.3 V, with a scan rate of 0.001 V/s. The surface area exposed to the solution was 0.38 cm². Then, according to the obtained polarization diagrams, anodic and cathodic Tafel slopes (β_a and β_c , respectively) were plotted; the necessary data were extracted via Tafel extrapolation and the corrosion potential (E_{corr}) and corrosion current density (I_{corr}) were obtained along with the corrosion rate. The size of the round samples used was $\varnothing 12 \times 4$ mm.

3. Results and Discussion

3.1. Microstructural Analysis

The WIP-C1 powder consisting of Ni and CrC powders was deposited on AISI 52100 steel. An SEM image of the substrate surface is presented in Figure 2a. Cross-sections of the substrate–deposit interface at different magnification levels are presented in Figure 2b–d. Figure 2c,d show that both the particles and the substrate surface were deformed upon multiple particle impacts. This substrate has high hardness which amplifies the degree of deformation of the particles, allowing for successful deposition, resulting in dense and tightly bonded coatings on the substrate surface. Figure 2b shows that the deposited surface is flat and free of cracks, both in the coating material and in the particles within the coating material. The dimensions of the deposited layer are approximately between 430 μm and 465 μm .

Figure 3 shows the coating surface at two different magnifications. This figure shows that particles of different sizes impacted the surface and the small particles that propelled at higher velocities interlocked better with the rest of the particles on the substrate surface. The dimensions of the particles were approximately between 17 μm and 66 μm (Figure 3a), and after EDS tests, the chemical composition was composed of compounds from Ni, Cr, C, and O (Figure 3c). Table 5 shows the mass percentage of the component elements of the deposited layer after EDS tests.

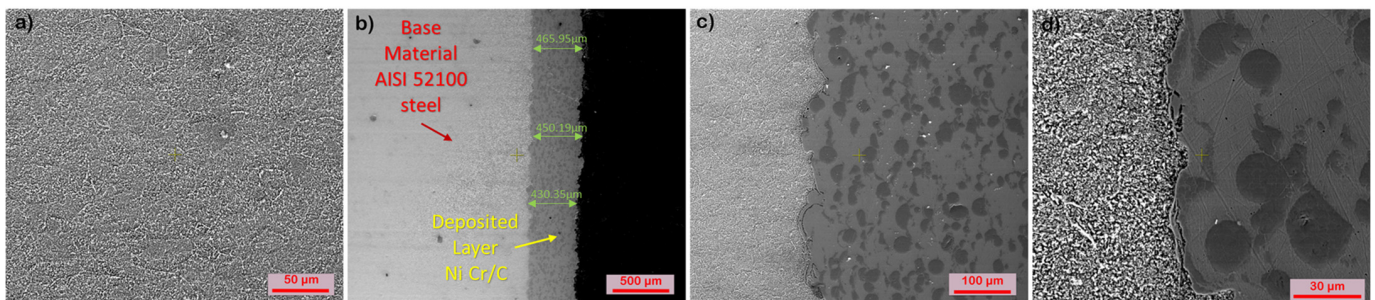


Figure 2. SEM images: (a) 1000 $\times$ base material; (b) 100 $\times$ and the cross-section of the coating; (c) 500 $\times$; (d) 2000 $\times$.

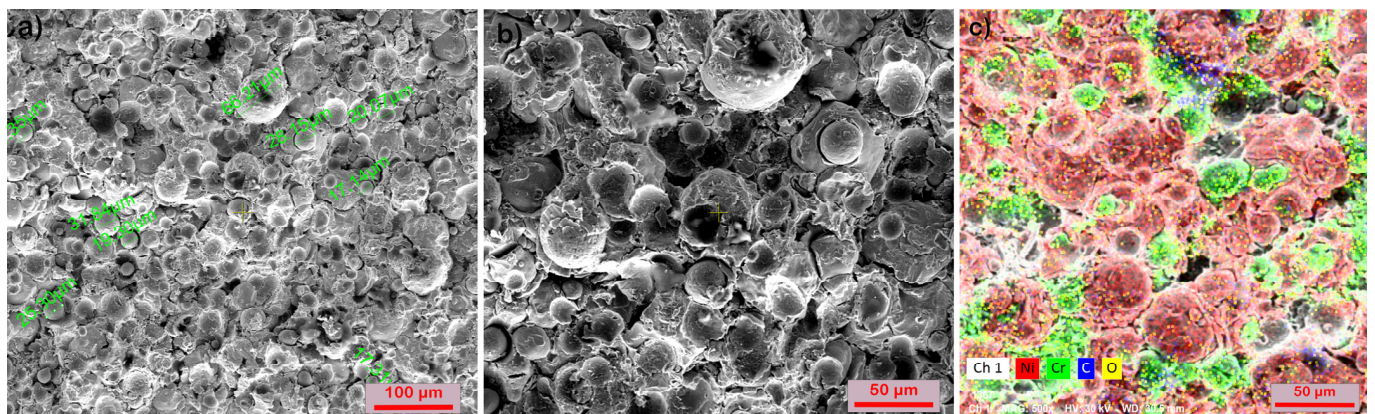


Figure 3. SEM images of the surface morphology of deposits: (a) 500 $\times$; (b) 1000 $\times$. (c) EDS mapping surface.

Table 5. Chemical composition after EDS tests of the surface coated with WIP-C1 powders.

Composition (wt%)	Cr (%)	O (%)	Ni (%)	C (%)
WIP-C1	22.25	3.44	57.65	16.64

3.2. Analysis of Mechanical Properties

To evaluate the Rockwell hardness and Young modulus, five microindentation tests were carried out on four coated 52100 steel samples and one uncoated, for 20 N vertical loading, and the final data are presented in Table 6. The coated samples exhibit greater hardness values compared to the base material, indicating enhanced wear resistance.

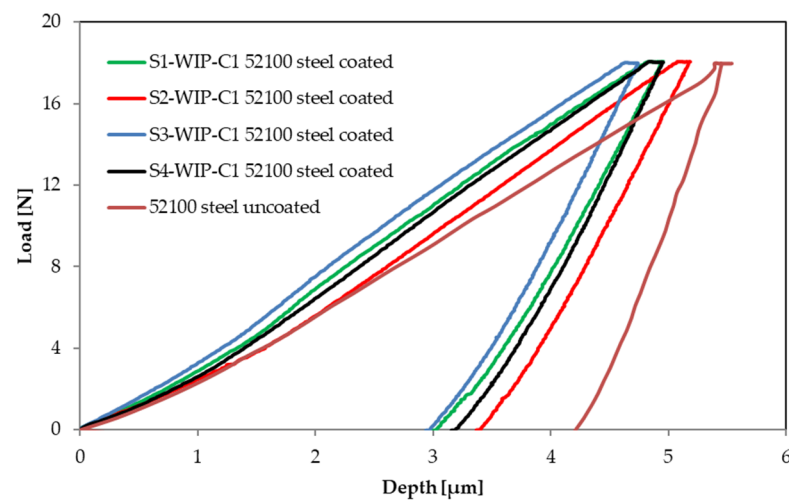
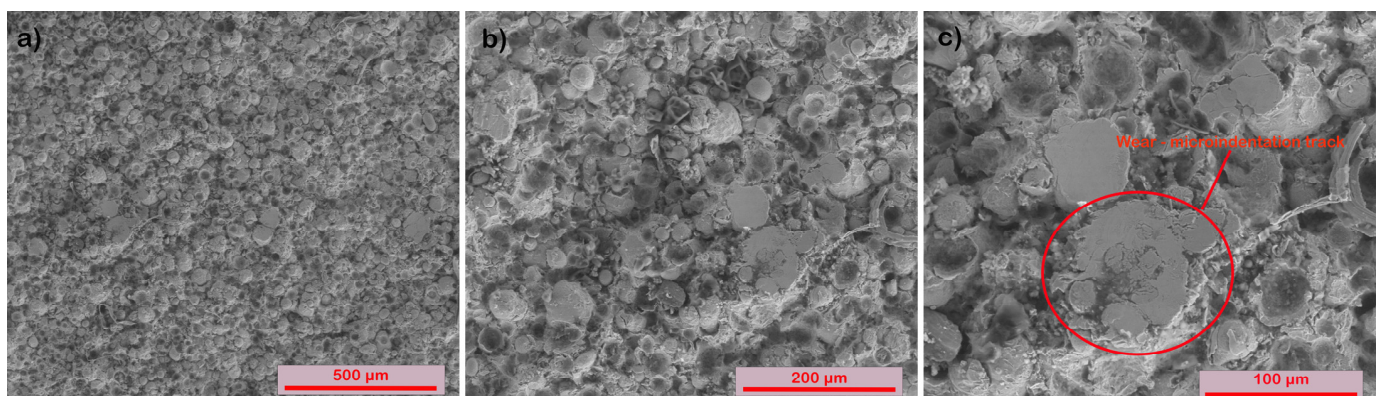
The graphs resulting from the microindentation tests show the specific load and the penetration depth; Figure 4 shows comparative graphs for the coated and uncoated samples, with a load of 20 N.

According to the indentation test procedure, at the beginning and towards the end of the indentation test, the loading force was maintained at 10% of the maximum value, with the 10% value being automatically subtracted by the data processing software.

In Figure 5, typical microindentation imprints at 20 N obtained by SEM are shown. These images show that following the indentation and deformation of the particles, the surface is smooth, and no microcracks are visible in the deposited layer.

Table 6. Data obtained after performing microindentation tests.

Sample		1	2	3	4	Uncoated
Load [N]				20		20
Rockwell Hardness [HRC]	1	69.60	65.80	72.23	70.44	49.31
	2	68.36	61.74	72.97	59.37	50.96
	3	73.87	68.07	67.19	65.95	47.28
	4	70.72	69.23	70.97	71.64	52.45
	5	63.88	64.79	76.26	69.03	48.59
	Av	69.29	65.92	71.92	67.29	49.72
Young modulus [GPa]	1	155.47	152.82	162.22	166.88	199.86
	2	154.38	159.67	169.53	153.40	208.04
	3	160.55	160.00	165.44	162.03	221.24
	4	155.92	157.04	160.25	165.28	211.39
	5	166.72	160.88	168.86	163.77	209.69
	Av	158.61	158.08	165.26	162.27	210.04

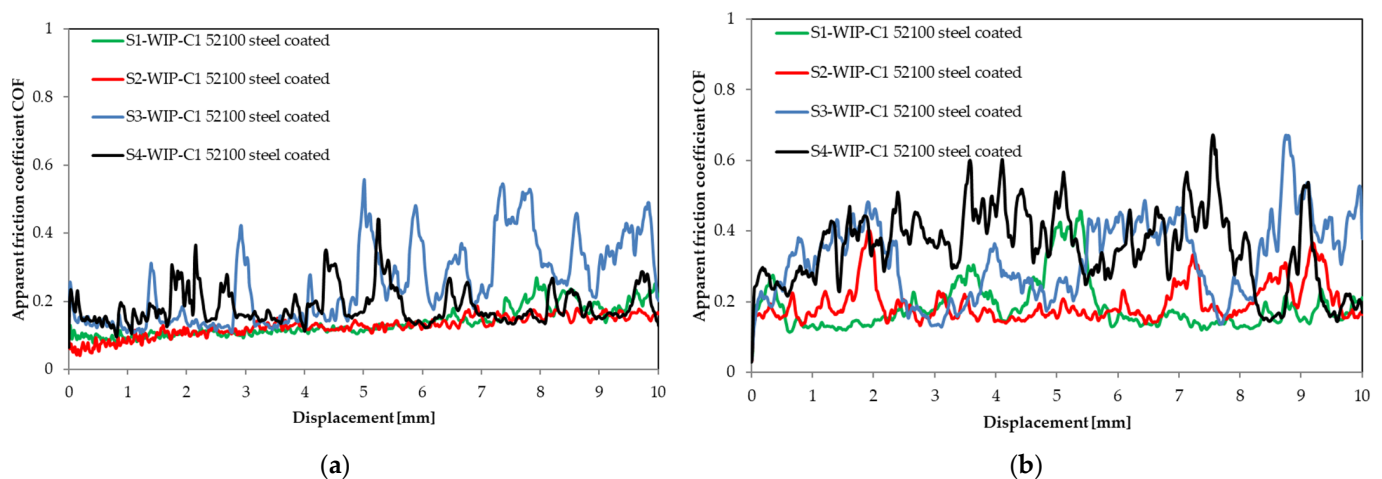
**Figure 4.** Microindentation graphs with the applied force and the depth of penetration for a load of 20 N.**Figure 5.** SEM images of microindentation prints: (a) 200×; (b) 500×; (c) 1000×.

To determine the microscratch resistance, four samples were subjected to testing at a linearly increasing load from 1 N to 10 N and a constant load of 10 N. The data obtained after the tests are presented in Table 7.

Table 7. The data obtained from microscratch resistance tests following the ASTM D7187 standard.

Samples	1	2	3	4
Linear load [N]				
COF	0.255	0.213	0.484	0.379
Constant load [N]				
COF	0.329	0.347	0.559	0.615

Figure 6 shows the graphs obtained after the tests performed, where you can see the variation in the apparent friction coefficient COF with Y displacement, many peaks, and average values above 0.2. The SEM images also show that the samples exhibit good resistance to microscratches. This figure shows the graphs resulting from linear load and constant load tests (sample 1 green line; sample 2 red line; sample 3 blue line; sample 4 black line), with (a) samples for linear load at 10 N and (b) samples for constant load at 10 N. For linear loading, the force was increased from 1 N to 10 N in 60 s, which was the test time frame for recorded data.

**Figure 6.** Graphs obtained after the tests to determine the resistance to microscratches: (a) samples for linear load at 10 N; (b) samples for constant load at 10 N.

From the SEM images shown in Figure 7, at $200\times$ it can be seen that for linearly increasing loading, the width of the microscratch track increases with increasing loading, and for constant loading the width of the microscratch track remains the same, approximately $260\text{ }\mu\text{m}$. The $500\times$ and the $1000\times$ SEM images also show that the microscratch track is only on the coating material, and the microscratch blade does not touch the base material. We can see that the microscratch blade only removes the large asperities of the coating material surface, indicating very good microscratch resistance of the coatings.

To determine the friction coefficient, tests were performed in both linear and rotational sliding modes. In the linear mode, two coated samples were subjected to 10 N, two coated samples were subjected to 20 N normal force, and two uncoated samples were subjected to applied forces of 10 N and 20 N. When performing the tests, the uncoated samples were tested for 15 min and the coated samples were tested for 60 min with a linear speed of 10 mm/s. The data obtained from the tests are presented in Table 8 and the graphs resulting from the testing are presented in Figure 8. From Figure 8c, we can observe that the steady state of friction coefficient variation was achieved faster for uncoated samples than the coated samples, so the test time was shorter.

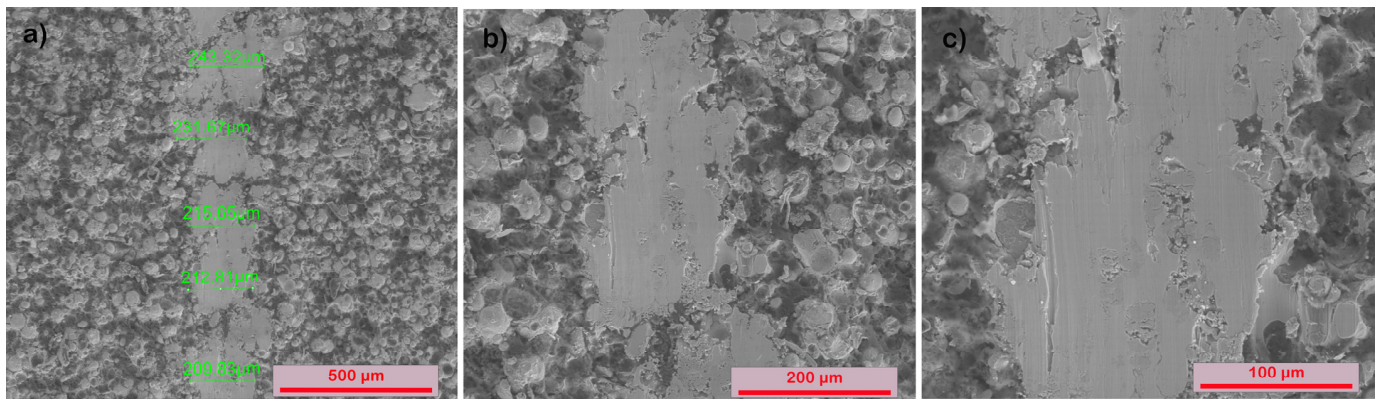
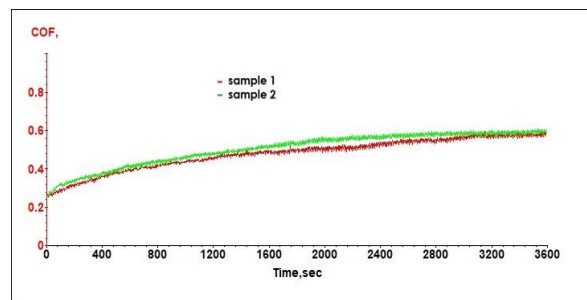


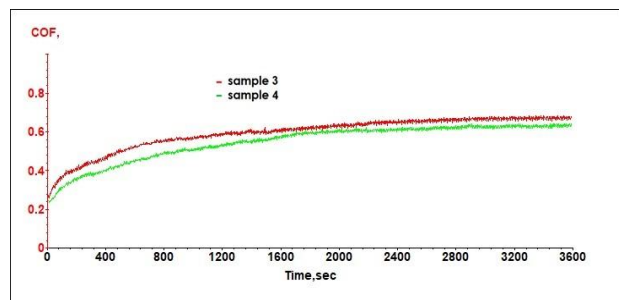
Figure 7. SEM images of microscratch tests: (a) 200×; (b) 500×; (c) 1000×.

Table 8. The coefficient of friction data obtained through linear sliding tests according to the ASTM 132 standard.

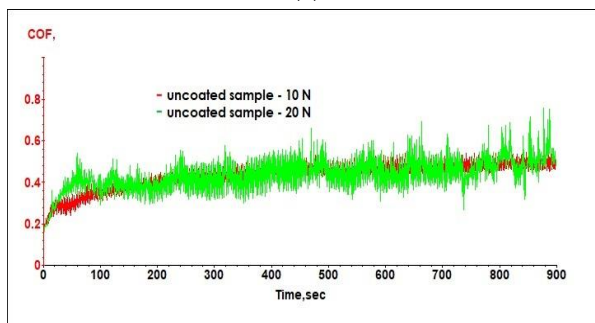
Samples	1	2	3	4	Uncoated 1	Uncoated 2
Load	10	10	20	20	10	20
COF	0.572	0.594	0.653	0.648	0.434	0.435



(a)



(b)



(c)



(d)

Figure 8. Graph results for the determination of the friction coefficient in linear sliding mode: (a) coated samples 1 and 2 at 10 N for 60 min; (b) coated samples 3 and 4 at 20 N for 60 min; (c) uncoated samples at 10 N and 20 N for 15 min; (d) the sample subjected to the test.

The rotational sliding tests consisted of testing two coated samples subjected to an applied force of 10 N, two coated samples subjected to an applied force of 20 N, and an uncoated sample subjected to an applied force of 10 N. The uncoated sample was tested for 15 min and the coated samples were tested for 60 min with a linear speed of 94.24 mm/s. The data obtained from the tests performed are presented in Table 9 and the resulting graphs can be seen in Figure 9. We can see from Figure 9c that the steady state of friction

coefficient variation was obtained faster for the uncoated samples than it was for the coated samples, which means that the test duration was shorter for the uncoated samples.

Table 9. The coefficient friction data obtained through rotational sliding tests.

Samples	1	2	3	4	Uncoated
Load	10	10	20	20	10
COF	0.614	0.643	0.668	0.687	0.196

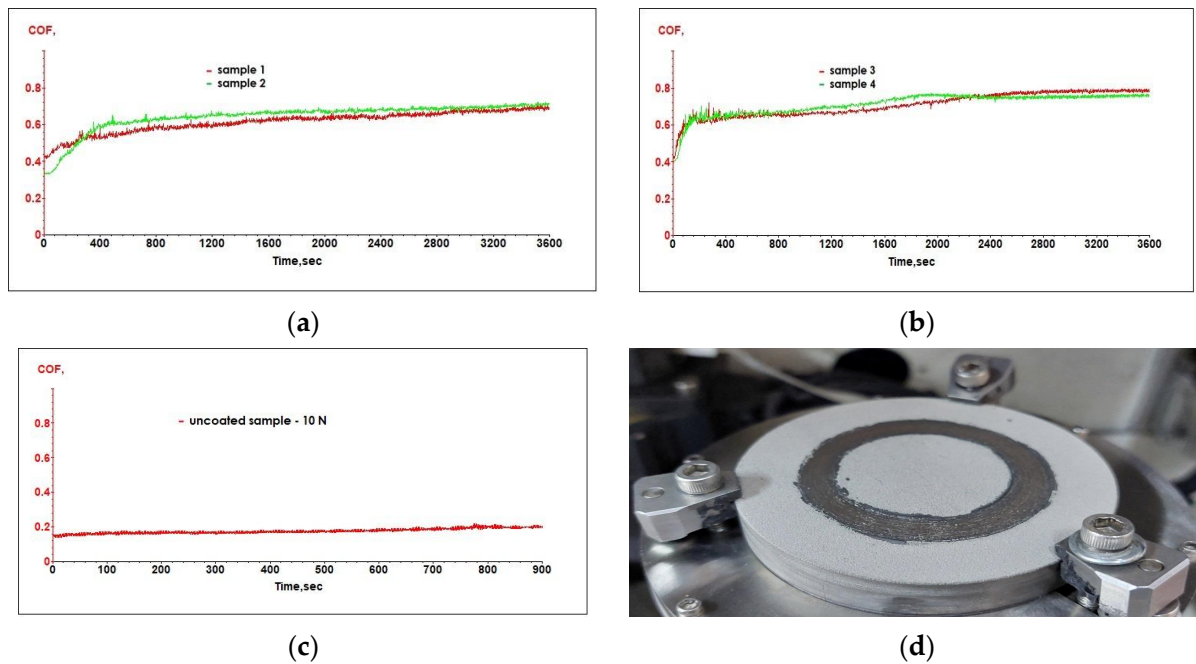


Figure 9. Graph results for the determination of the friction coefficient by rotational movement: (a) coated samples 1 and 2 at 10 N for 60 min; (b) coated samples 3 and 4 at 20 N for 60 min; (c) uncoated sample at 10 N for 15 min; and (d) the sample subjected to the test.

At the beginning of the friction tests, a short transition period was observed as the apparent contact area increased until it reached a steady state and the larger peaks of the coating surface asperities were removed. The last observation explains the high values of the friction coefficient, between 0.4 and 0.7. Also, from the SEM images shown in Figure 10, it can be seen that the peaks of asperities were removed and the particles remaining on the sliding track acted as an abrasive material leading to high values of the friction coefficient.

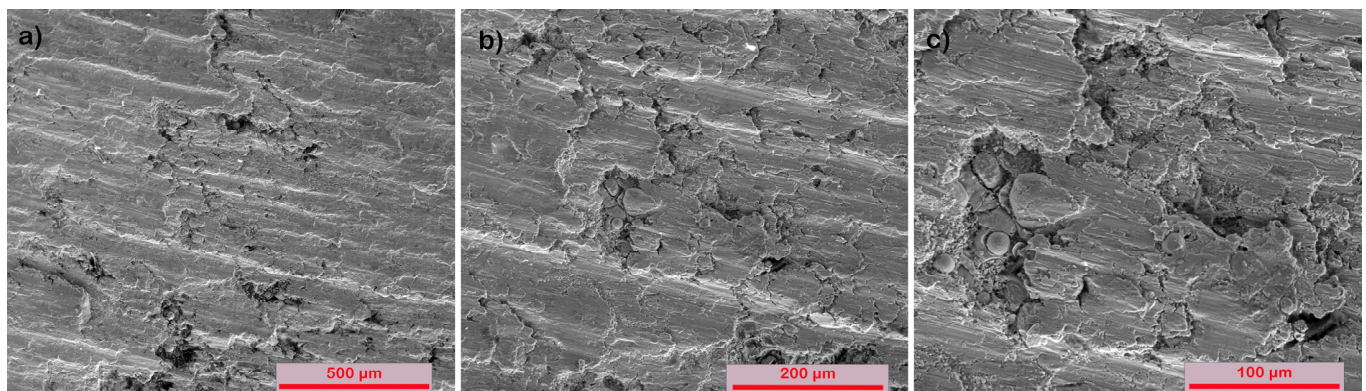


Figure 10. SEM images of the sample surface after the friction tests: (a) 200×; (b) 500×; (c) 1000×.

3.3. Corrosion Resistance

Electrochemical corrosion processes in the coatings were investigated using potentiometric testing methods and electrochemical impedance spectroscopy. To obtain instantaneous information on corrosion rates, linear polarization resistance and Tafel extrapolation techniques were used. A significant improvement in the corrosion resistance can be observed in the coated samples. Figure 11 shows the potentiodynamic polarization plots for the coated and uncoated substrates in acid rain solution $\text{HNO}_3\text{--H}_2\text{SO}_4$. As can be seen from the plots, the coated sample shows lower I_{corr} and higher E_0 than the uncoated sample due to the electrolyte being blocked by the coating. As shown in Table 10, the I_{corr} value (18.37 mA) for the substrate is the highest. This difference is also confirmed by the corrosion rate value of 88.48 mpy for the uncoated substrate compared to 18.95 mpy for the coated substrate. This lower rate is due to the condition of the surface of the sample, i.e., a flat surface without cracks, confirmed through microstructural analysis, as mentioned above. Due to the nature of the coating materials, Ni/CrC coatings exhibit excellent chemical stability and corrosion resistance in the most aggressive environments.

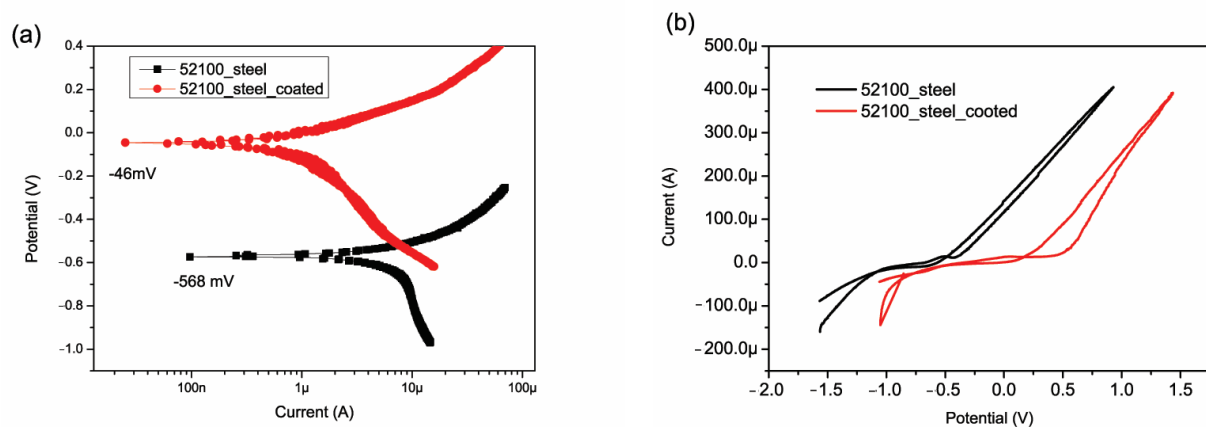


Figure 11. (a) Tafel plots; (b) cyclic polarization.

Table 10. Electrochemical parameters obtained from Tafel plots.

Sample	E_0 (mV)	I_{corr} (mA)	Corrosion Rate (mpy)
52100 steel substrate	−568	18.37	88.48
Ni/CrC coated 52100 steel substrate	−46	3.9	18.95

More complete information on the electrochemical processes at the electrodes or in solution can be obtained using the cyclic polarization curve. Cyclic polarization plots are presented in Figure 11b. The anodic curves overlapped with difficulty over the cathodic curves for both samples, as is characteristic of generalized corrosion, but at the same potential the current density for the coated sample was lower than for the uncoated sample. It can be observed that the breakdown potential for the coated sample was around 0.6 V and for the uncoated sample it was around −0.4 V, indicating more electronegativity.

Figure 12 shows the Bode and Nyquist plots obtained in acid rain solution $\text{HNO}_3\text{--H}_2\text{SO}_4$. The fit parameters are listed in Table 11. The recorded impedance data agree with the model shown in Figure 12. R_s corresponds to the uncompensated resistance between the reference electrode and the working electrode, CPE refers to the passive surface capacity, and R_p corresponds to the passive surface resistance. CPE impedance is defined by

$$Z_{\text{CPE}} = \frac{1}{Q(j\omega)^n} \quad (1)$$

where Q is a constant proportional to the active area ($\langle Q \rangle = \Omega^{-1} \text{ sn/cm}^2 \equiv S \cdot \text{sn/cm}^2$), ω is the angular frequency ($\omega = 2\pi f$, where f is the frequency of applied alternating current), and j is the imaginary number ($j = (-1)^{1/2}$) [34].

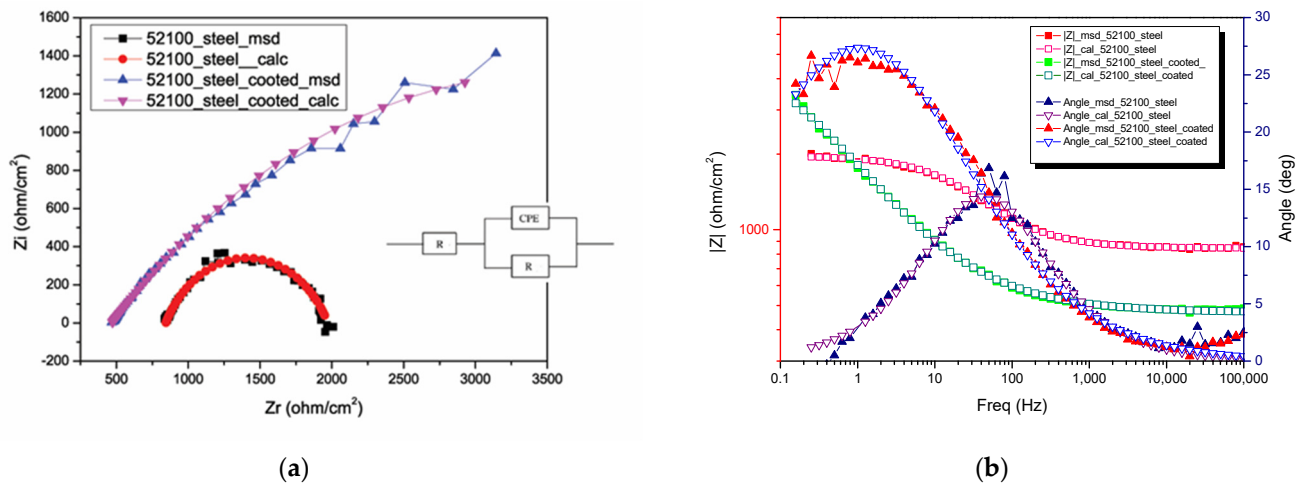


Figure 12. (a) Nyquist and (b) Bode plots for 52100 and 52100 coated steel.

Table 11. Simulated parameters of EIS equivalent circuit fitting.

Sample Parameter	R_s (ohm·cm ²)	CPE (S·s ⁿ /cm ²)	n	R_{ct} (ohm·cm ²)
52100 steel substrate	843	0.0000276	0.8	1130
Ni/CrC coated 52100 Steel substrate	469	0.000233	0.5034	6301

The capacitance data obtained from EIS can be used to determinate the compact passive film, which is related to the resistance and capacitance of the passive film. The value obtained for R_{ct} increases from 1130 (ohm·cm²) to 6301 (ohm·cm²) for the 52100 steel coated sample. This can be attributed to the protection provided by the coating. The Bode diagram shows the existence of a single maximum on the curve (phase angle as a function of applied signal frequency), implying the existence of a single relaxation time constant. In general, three frequency regions referring to high, medium, and low values can be distinguished from the impedance spectrum. The high frequency range of impedances at frequencies above 103 Hz highlights the values of the solution resistance (R_{sol}). In the Bode plot, the impedance at medium frequencies represents the response of the surface layer, while at the lower frequency limit, the process information is related to the substrate response at the electrolyte interface.

The high impedance values (on the order of 106 $\Omega \cdot \text{cm}^2$) obtained from the middle and low frequencies for all coated samples suggest high corrosion resistance in electrolyte solution. The values obtained for the maximum phase angle open up three frequency ranges (0.1–100 Hz), indicating a capacitive response for all samples and suggesting the presence of a surface layer acting as a barrier.

3.3.1. Spectrochemical Analysis and Basic Material

At the level of the metal, it can be observed that the surface is corroded, showing chemical corrosion compounds that seem to be different to those of steel, being assessed as oxides and carbonates. The appearance of the compounds is the result of the interaction between the metal and the electrolyte solution. Corrosion is created on the entire surface of the sample, in general, and through several investigations its nature can be established. Figure 13 shows SEM images of the surface after open circuit potential (OCP), linear and cyclic potentiometry (LP and CP), and electro-impedance spectroscopy (EIS).

Table 12. Surface’s chemical composition after electrochemical tests.

Element	At. No.	Netto	Mass [%]	Mass Norm [%]	Atom [%]	abs. Error [%] (1sigma)	rel. Error [%] (1sigma)
Iron	26	136520	94.03	92.96	81.88	2.41	2.57
Oxygen	8	1056	2.55	2.52	7.75	0.81	31.86
Nitrogen	7	374	2.41	2.38	8.38	0.87	35.92
Chromium	24	3704	1.50	1.48	1.40	0.08	5.341
Manganese	25	1188	0.66	0.65	0.58	0.07	10.79
Sum			101.15	100	100		

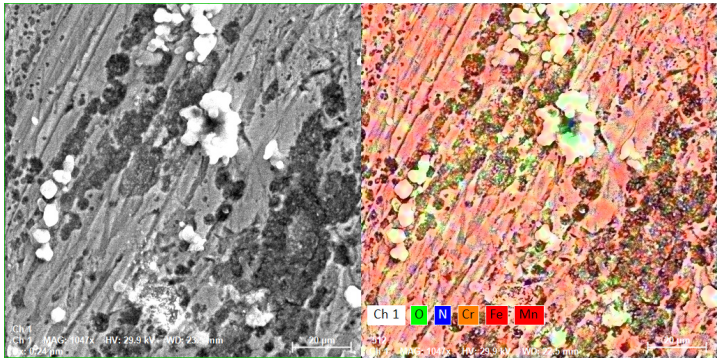


Figure 15. Elemental distribution on the corroded surface (all elements).

3.3.2. Spectrochemical Analysis and Coated Material

Through micro-scale electro-corrosion tests, it was observed that the surface of the Ni/CrC coating was intact. Only a few compounds were present on the coating surface that originated from the electrolyte solution, or that were a result of the interaction between the solution and the metal–ceramic system. Figure 16 shows SEM images of the surface after open circuit potential (OCP), linear and cyclic potentiometry (LP and CP), and electro-impedance spectroscopy (EIS).

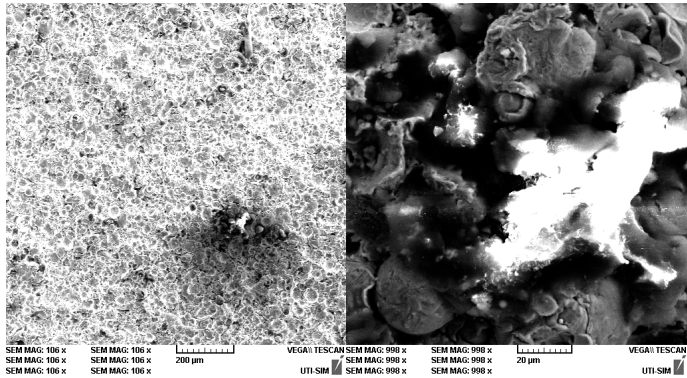


Figure 16. SEM images of the surface obtained via open circuit potential (OCP), linear and cyclic potentiometry (LP and CP), and electro-impedance spectroscopy (EIS).

On the surface of the sample, on the micro-scale, few compounds can be observed. By analyzing the chemical composition, in addition to the elements that make up the deposited layer (Cr, Ni, Si, and C), other elements such as nitrogen, oxygen, and sulfur were identified in small quantities, as seen in Figure 17. Unlike the material of the base of the tested sample, it can be appreciated that the deposited superficial layer shows significantly lower oxidation. Table 13 shows the chemical composition of the surface after electrochemical tests.

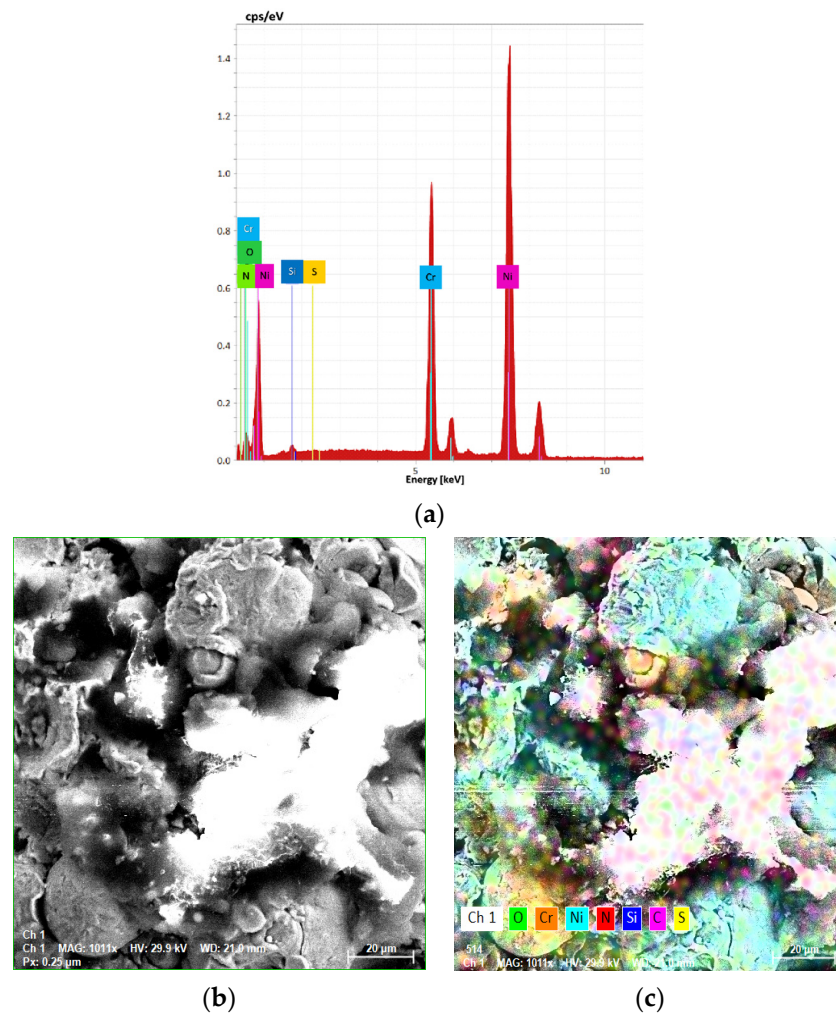


Figure 17. The energy spectrum of the elements identified on the corroded surface: (a) EIS test; (b,c) elemental distribution on the corroded surface (all elements).

Table 13. Chemical composition of the surface after electrochemical tests.

Element	At. No.	Netto	Mass [%]	Mass Norm [%]	Atom [%]	abs. Error [%] (1sigma)	rel. Error [%] (1sigma)
Nickel	28	84670	80.83	69.62	56.99	2.05	2.53
Chromium	24	49486	27.16	23.39	21.62	0.75	2.76
Nitrogen	7	602	4.68	4.03	13.84	1.36	29.05
Oxygen	8	729	2.25	1.94	5.83	1.12	49.55
Silicon	14	1060	1.11	0.96	1.64	0.11	9.94
Sulfur	16	68	0.04	0.04	0.06	0.02	64.32
Sum			116.09	100	100		

4. Conclusions

This work showed that Ni/CrC forms a stable coating on 52100 alloy steel when deposited by the cold spray technique.

The microstructural analysis showed that the coatings are dense and tightly connected to the surface of the substrate, and through the collision and simultaneous deformation between the particles and the base substrate, the ceramic particles are deeply inserted, achieving this strong connection through mechanical interlocking.

Microindentation tests led to obtaining maximum hardnesses of 70 HRC with a Young modulus of 165.26 GPa for a load of 20 N of the coated samples. For the uncovered samples, a value of 50 HRC with a Young modulus of 210.04 GPa for the 20 N load was achieved.

The microscratch testing showed that the Ni/CrC coating has good resistance to sliding, as the friction coefficient showed large variability along the sliding path with average values higher than 0.2. From the SEM images, it can be seen that the width of the scratch track is approximately 260 μm ; the scratched area is only on the coating material; and the base material is not affected. It can be observed that the blade removes only the large asperities of the coating, which confirms the achievement of good resistance to microscratches.

Following the determination of the coefficient of friction, a short transition period was observed at the beginning of both tests as the apparent contact area increased until it reached a steady state and flattened the large peaks of the coating asperities.

Linear potentiometry and EIS tests confirmed very good corrosion resistance for the coated samples. The passive, stable, non-porous, and non-cracked characteristics of the coating were confirmed by the low corrosion rate values and the high strength of the coating. The fact that the Ni/CrC coating's surface was still intact after the corrosion tests serves as additional confirmation of this.

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