

Article

Surface Characteristics of Vacuum-Plasma-Sprayed Reinforced Stainless Steel Coatings with TiC Particles

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Abstract: Fe-based coatings reinforced with TiC particles exhibit outstanding properties and are widely recognized as highly promising coatings or components with superior performance. In the present study, composite materials using a powder mixture of AISI 316 stainless steel and TiC particles were deposited by vacuum plasma spraying onto a S235 low alloyed steel substrate. The coating and the base material were characterized in terms of microstructure and surface properties. The metallographic analysis of the deposited coating revealed the uniform distribution of the TiC into the stainless steel matrix. The results show that the presence of tungsten carbide particles improved the hardness and tribological properties of the composite coating compared with the S235 base material. The wear resistance of the coating was approximately seven times higher than that of the low alloyed steel substrate. The electrochemical corrosion resistance of the coating in chloride media was much higher than that of the base material.

Keywords: vacuum plasma spraying; stainless steel; TiC particles; corrosion; wear; hardness; microstructure



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1. Introduction

Surface engineering is a branch of materials science and engineering focused on modifying the surface properties of materials to enhance their performance, durability and functionality. It involves the application of various techniques and processes to alter the surface composition, structure and properties of materials while leaving the bulk properties largely unchanged. It is considered an enabling technology, particularly in the manufacturing industry, where it is used to meet the demands for enhanced friction, wear and corrosion resistance [1–3].

The techniques related to surface engineering have a wide range of applications in the automotive industry, aerospace, electronics, cellulose and paper industry, maritime shipbuilding industry, construction of thermo- and hydro energy equipment, orthopedics, dentistry, etc. [2–4]. Among these, plasma spraying is a key technique that has been used for over four decades to deposit metallic materials, ceramic materials and even composite materials. It has been used particularly in the aerospace and automotive industries due to its ability to produce high-performance coatings [5].

Iron-based thermally sprayed coatings have been a favorable choice as an alternative to tungsten-carbide-based coatings, primarily owing to their eco-friendly characteristics and cost effectiveness. These coatings are employed across various applications, especially when there is a need for both wear and corrosion resistance [2,3]. These phenomena predominantly manifest on the material surface and are the primary cause of material

failure [4]. The specific composition and properties of the coatings can be tailored to meet the requirements of each application, making them a popular choice for surface protection and enhancement. Therefore, implementing protective coating methods on material surfaces is crucial [6].

Research on iron-based thermally sprayed coatings has shown promising results in various applications. Consequently, several stainless steel alloys have shown good corrosion properties, especially when they were exposed to cavitation erosion conditions [7–10]. De et al. [11] in their research demonstrated that 304 stainless steel stands out as an outstanding material resistant to cavitation, offering significant application potential across various fields. This is attributed to its favorable characteristics, including good plasticity, high corrosion resistance and cost effectiveness. Also, the martensitic steels [3,9,10] have demonstrated outstanding resistance to cavitation erosion, and this performance was closely associated with their hardness. Nevertheless, in numerous instances, softer austenitic or dual-phase steels have proven to be even more advantageous, primarily because of their superior strain hardening properties [3,12–16]. Moreover, Milanti et al. [17] demonstrated relatively favorable cavitation erosion performance for a high-velocity oxygen fuel (HVOF)-sprayed Fe-Cr-Ni-C-B alloy coating, and Yupin et al. [18] observed similar results for an HVOF-sprayed Fe-Cr-Si-B-Mn coating. Also, plasma spraying has been used to create stainless steel coatings with enhanced microhardness and tensile adhesion strength. Bhat et al. [19] found that increased quench rates during the plasma spraying of martensitic stainless steel result in finer grain and second-phase distributions, as well as enhanced microhardness and adhesion strength. Zhao and colleagues [20] deposited dense nitrogen alloyed austenitic steels using atmospheric plasma spraying (APS) and high-velocity oxy-fuel (HVOF) methods and tested their surface properties. The results showed good corrosion resistance and microhardness properties for the coatings deposited by the HVOF process. By atmospheric plasma spraying (APS) and vacuum plasma spraying (VPS), Zhai et al. [21] manufactured Fe-based ($\text{Fe}_{46.8}\text{-Mo}_{30.6}\text{-Cr}_{16.6}\text{-C}_{4.3}\text{-B}_{1.7}$) metallic glass coating layers and studied their microstructure and corrosion properties. The results showed that VPS coatings had a microstructure with higher corrosion resistance compared with the layer produced by the APS process. Xiao and colleagues [22] investigated the microstructure and wear behavior of plasma-sprayed FeCoNiCrMn high-entropy alloy coatings by varying the H_2 flow rate. They obtained improved wear resistance from higher H_2 flow rates because of the cohesive strength among splats and the increase in oxides. Tsunekawa et al. [23] and Ding et al. [24] explored the application of plasma spraying for iron-based coatings. The first investigated the addition of solid lubricants, such as boron nitride (h-BN), to improve the tribological properties of the deposited coatings. The results obtained showed lower coefficients of friction and better wear resistance for the coatings prepared with h-BN particle additions. Ding examined the cutting process of plasma-sprayed iron-based alloys. These studies collectively highlight the potential of vacuum plasma spraying for enhancing the properties of iron-based coatings.

The thermal spraying of Fe-based coatings has developed rapidly in the past two decades. The process of thermal spraying, using either gas combustion or electrical discharge, involves heating the feedstock particles to a molten or semi-molten state. Subsequently, these particles are sprayed onto the substrate through flame flow or compressed air. As the particles hit the substrate, they flatten and solidify, forming stacked splats that ultimately create coatings [25–27].

Considerable research has been conducted on the thermal spraying of coatings based on iron, with particular attention given to analyzing their microstructure, magnetic characteristics and stability [28]. The use of a newly developed thermal spraying technique has enabled the production of amorphous Fe-based coating films with high hardness and corrosion resistance [29]. These coatings have been found to exhibit wear, corrosion and high-temperature-resistant properties, with the performance depending on the coating structure and the presence of reinforcement particles and alloying elements [30]. However,

the wear behavior of these coatings, particularly in power generation plants, has also been investigated, revealing the presence of various phases and inhomogeneities [31].

Classification of thermal spraying processes includes various processes based on the thermal energy source and the kinetic energy of the sprayed materials, such as electric arc spraying (AS), plasma spraying (PS) and high-velocity oxy-fuel (HVOF) spraying. Within the PS category, there is a subdivision into atmospheric plasma spraying (APS) and vacuum plasma spraying (VPS) based on the operating pressure range [32]. APS involves coating formation in an open-air environment, making the metallic coating susceptible to oxidation and a reduction in density due to the formation of pores [32,33]. In contrast, VPS helps prevent oxidation of the coating and enhances coating density, as molten droplets experience low atmospheric pressure and high velocity.

Over the past decades, there have been many methods used to improve the strength, toughness and hardness of steels. Tool steels and high-strength steels employ metallurgical strengthening mechanisms, such as phase transformations inducing strain hardening or high solid solution concentrations, leading to solid solution strengthening, dispersion strengthening and precipitation hardening. Heat treatment has a significant positive effect on the properties of these steels. Stainless steel alloys like FeCrNi-based alloys use strengthening mechanisms such as solid solution precipitation, deformation twinning and the formation of ϵ -martensite and α -martensite phases [3,34].

Another method to improve the properties of iron-based coatings is developing composite coatings by reinforcing the metallic matrix with ceramic particles that combine the outstanding characteristics of both materials [4].

For this purpose, in the present study, stainless steel reinforced TiC composite coatings, which combine the good corrosion property of the metallic matrix and the wear stability of ceramic particles, were developed and deposited by vacuum plasma spraying onto the surface of a low alloyed steel substrate in order to improve the surface properties of the latter. A comprehensive investigation was carried out to analyze the coating substrate system regarding the microstructure, phase composition, hardness and wear and corrosion characteristics of the stainless-steel-based composite coating and of the substrate.

2. Materials and Methods

The raw materials used for the experiments included an FeCrNiMo powder (Amperit 377.065), which was mechanically dry mixed with 5 wt.% TiC particles (from Höganäs company, Düsseldorf, Germany) for 3 h. The commercial Amperit powder provided by H.C. Starck GmbH, Goslar, Germany had a chemical composition similar to AISI 316L stainless steel, as presented in Table 1.

Table 1. Nominal chemical composition of AISI 316L stainless steel.

Carbon (C)	0.029%
Silicon (Si)	0.92%
Manganese (Mn)	1.86%
Phosphorous (P)	0.034%
Sulfur (S)	0.013%
Chromium (Cr)	17.42%
Nickel (Ni)	12.51%
Nitrogen (N)	0.10%
Molybdenum (Mo)	2.21%
Iron (Fe)	Balance

This powder is recommended by the supplier to be used for manufacturing thermal sprayed coatings used for corrosion and cavitation protection. In order to increase the tribological properties of the coatings, 5 wt.% TiC was added by blending in the chemical composition of the iron-based coatings.

The composite powder mixture was sprayed by vacuum plasma spraying onto the surface of low alloyed steel S235. The chemical composition of the substrate, determined

by optical emission spectrometry (Thermo Arl QuantoDesk equipment, Burladingen, Germany), is presented in Table 2.

Table 2. Chemical composition of the S235 substrate.

Carbon (C)	0.18%
Manganese (Mn)	1.42%
Phosphorous (P)	0.037%
Sulfur (S)	0.021%
Silicon (Si)	0.042%
Iron (Fe)	Balance

Before deposition, the surface of the substrate was sandblasted and cleaned with acetone. The powder was heated and sprayed onto the substrate using robotic spray equipment from Sulzer Metco. By preliminary testing, the process's technological deposition parameters were determined:

- Arc current: 600 A;
- Gas mixture: Ar + 7% H₂;
- Feed rate: 25 g/min;
- Spraying distance: 200 mm;
- Pressure in the chamber: 135 Bar.

After the vacuum plasma spraying process, the coated sample was cut perpendicular to the surface, was metallographically prepared by grinding using abrasive paper of decreasing granularity and was polished with a diamond suspension until a scratch-free and mirror-like surface was obtained.

The coating and the substrate were investigated in terms of morphology and microstructure using the scanning electron microscope SEM Quanta FEG 250 equipped with EDAX analysis, and phase identification was carried out by X-ray diffraction techniques with Philips X'Pert Diffractometer, Panalytical, The Netherlands, equipped with monochromatic Cu-K_{α1} X-radiation. XRD diffraction measurements were carried out in 2 theta geometry in the range of 20° to 100° at a scanning rate of 1°/min. The operation was conducted at a voltage of 40 kV with a current intensity of 30 mA. The crystallographic identification of phases in the samples was performed using the JCPDS (Joint Committee on Powder Diffraction Standards) database.

The electrochemical corrosion of the substrate and the coating was measured in a 3.5% NaCl solution at room temperature using a three-electrode corrosion cell: a working electrode (sample), a reference electrode (saturated calomel electrode) and an auxiliary (platinum) electrode. The exposed surface in the corrosive media was 1 cm². The method was materialized by drawing the polarization curves of the tested materials. For the experiments, a corrosion cell equipped with an SP-150 galvanostat from Biologic Science Instruments was used. The samples were polarized in the anodic direction by varying the potential in the range of −2.5 V to 2.5 V. During the test, the corrosion potential and current density were measured. The recorded data were processed using the dedicated EC-Lab software, version 10.3, from Biologic. Before measuring the electrochemical parameters, the exposed surface in the corrosive media was grinded and polished until a metallic mirror was obtained.

The microhardness was measured in a cross section both for the coating and the S235 substrate using the tester equipment Zwick/Roell YHV_μ-S, Ulm, Germany. HV 0.3 measurements were recorded by applying a load force of 300 gf and a loading time of 10 s. The measurements were conducted at intervals of about 0.1 mm, and the average value calculated from three measurements was obtained for each depth.

The coating and the substrate were evaluated to determine their sliding wear properties. The evaluation of the wear resistance was made using the pin-on-disk method. The tribometer equipment TR-20 from DUCOM Materials Characterization Systems was set up for the following testing parameters: a normal load of 10 N, a relative velocity of 20 cm/s

between the tungsten carbide (WC) ball (diameter 6 mm) and the surface, and a testing distance of 1000 m. The trajectory followed a circular path with a diameter of 10 mm. The wear rates were determined by measuring the profile of the wear track.

3. Experimental Procedure

3.1. Powder Morphology and Coating Microstructure

The effectiveness of a coating is heavily influenced by its metallurgical properties, which can vary from one coating to another based on its unique microstructures. Therefore, comprehending the performance of any industrial component requires an examination of the properties exhibited by the distinct phases defining the material's microstructural composition. Typically, the microstructural condition of a coating serves as a distinctive indicator of the process through which the coating is applied.

Figure 1 presents an SEM micrograph of the powder, whose morphology was spherical with granulation, according to the supplier, between 15 and 45 μm . As can be seen, their equivalent diameter measurements during SEM analysis confirm the range size of the powder particles.

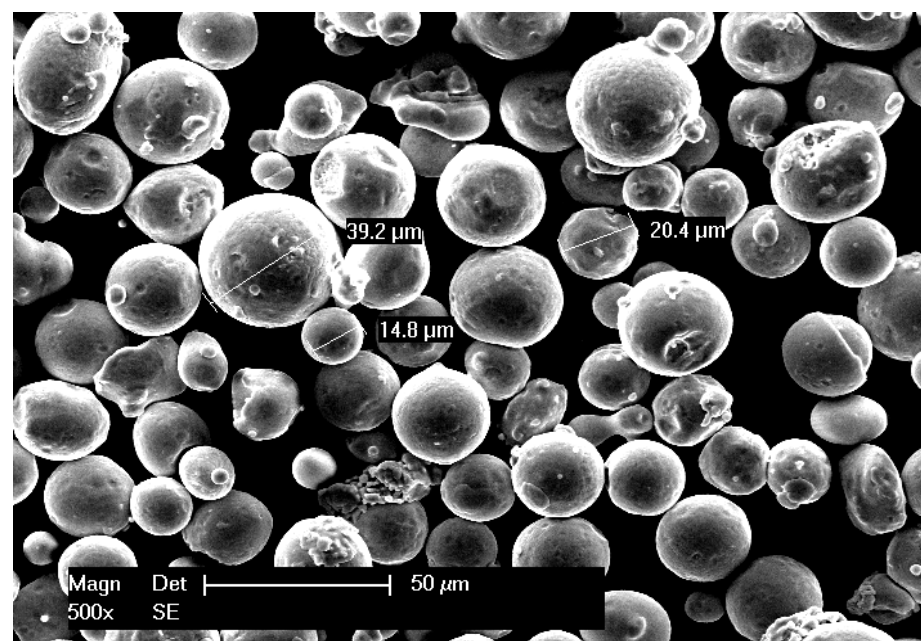


Figure 1. Morphology of the Amperit powder.

Figure 2 presents the SEM micrographs of the deposited coating using the composite powder stainless steel (SS) reinforced with TiC particles. The microstructure of the coating was mostly dense, and small numbers of homogenous pores were visible. The coating's thickness was about 900 microns (Figure 2a). Because the spraying process ran in a vacuum, no internal oxidation was observed. A good interface between the substrate and the coating was observed, which had a positive effect regarding the adherence of both system elements (Figure 2c). The TiC particles were uniformly distributed in the stainless steel matrix (Figure 2b). In the as-delivered state, the base metal had a microstructure consisting of separated ferrite between the critical temperatures $A_{r3} \dots A_{r1}$, perlite formed upon reaching the A_{r1} temperature and bainite formed at subcooling temperatures of approximately 350–400 $^{\circ}\text{C}$ (Figure 2d). With a carbon content of approximately 0.18%, the proportion of proeutectoid ferrite should be higher than that of perlite + bainite. The presence of the alloying element (Mn) in the chemical composition of the steel reduced the carbon concentration of the eutectoid point and increased the transformation stability of the subcooled austenite. Therefore, the obtained microstructure consisted of approximately 55%–65% ferrite (light color), approximately 10%–15% perlite (black color) and approximately 20%–35% bainite.

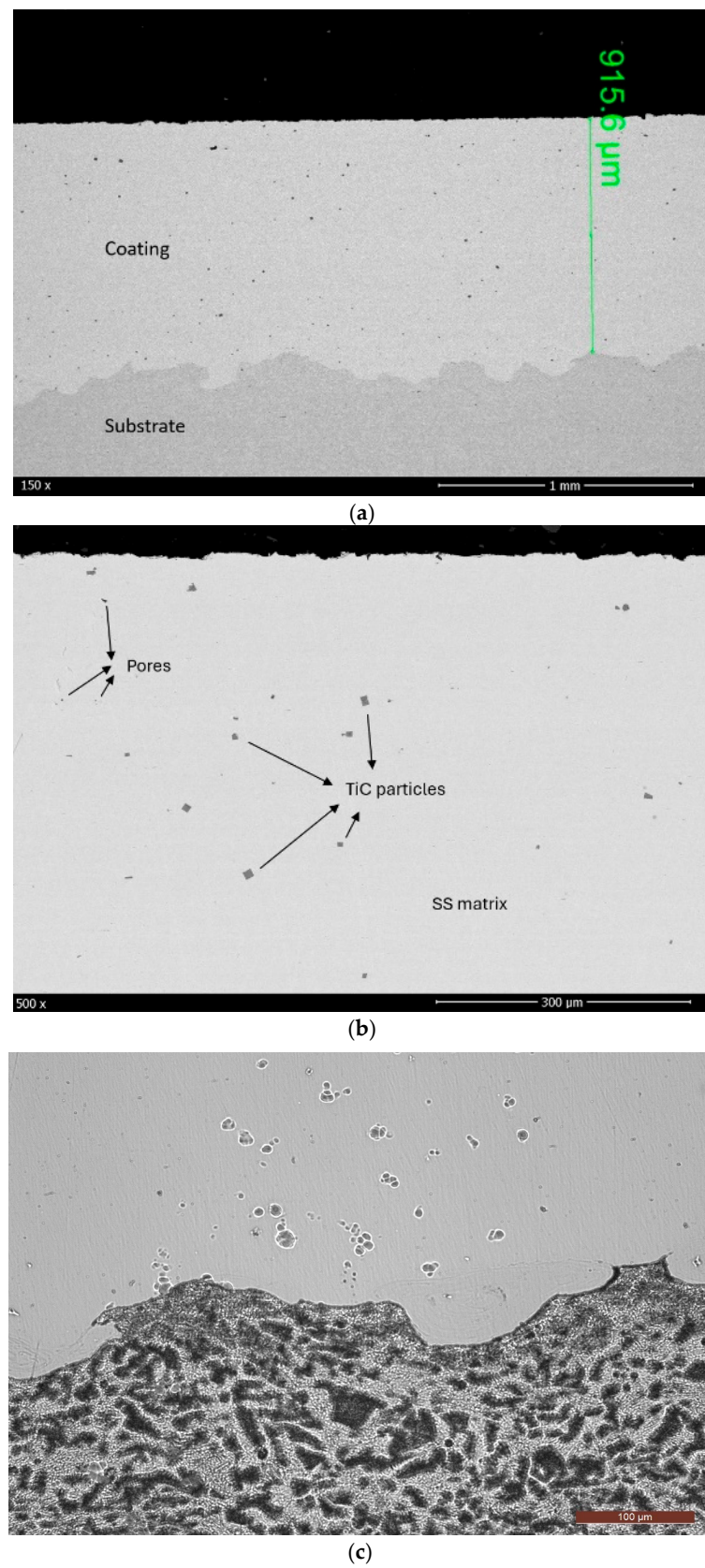


Figure 2. Cont.

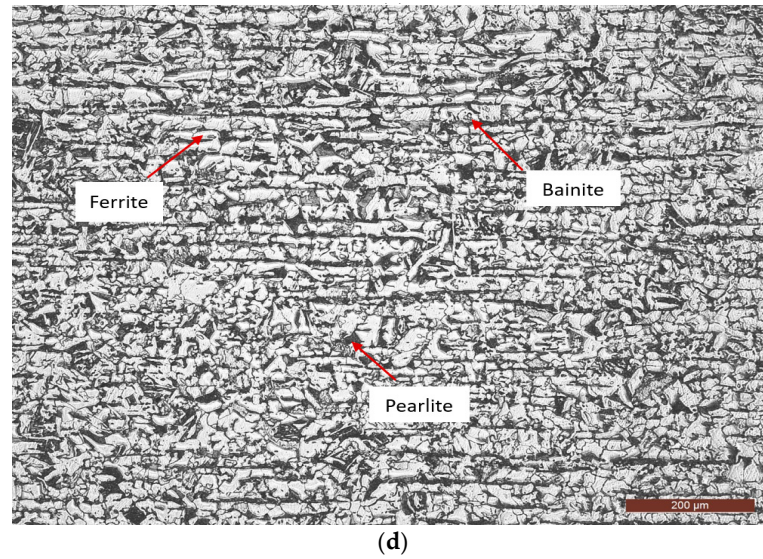
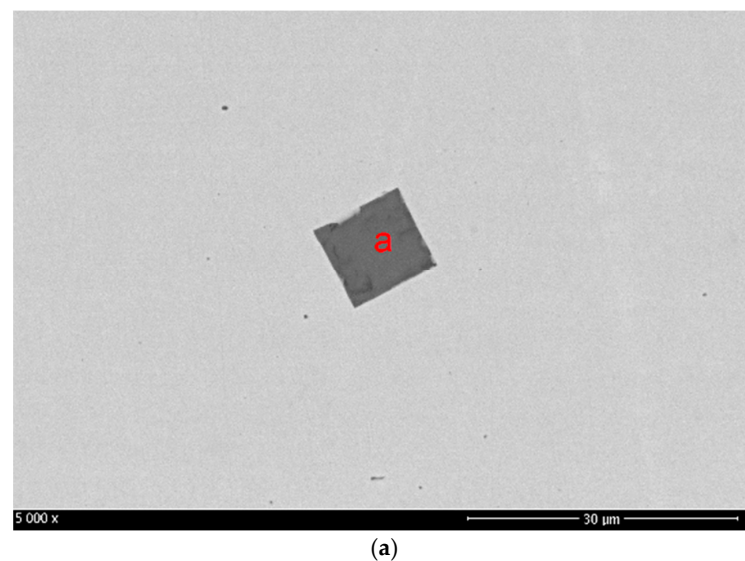


Figure 2. Microscopic analysis of the deposited SS + TiC composite coating: SEM micrographs (a) $\times 150$; (b) $\times 500$. Optical micrographs (3% Nital etched): (c) interface coating the substrate; (d) substrate.

In order to confirm the chemical composition of the used feedstock, Energy Dispersive X-Ray analysis (EDAX) was carried out. Figure 3 shows a TiC particle present in the metallic stainless steel composite matrix. The EDAX spectra from Figure 3 confirm the presence of the chemical elements in the deposited coating.



EDAX analysis of point **a**

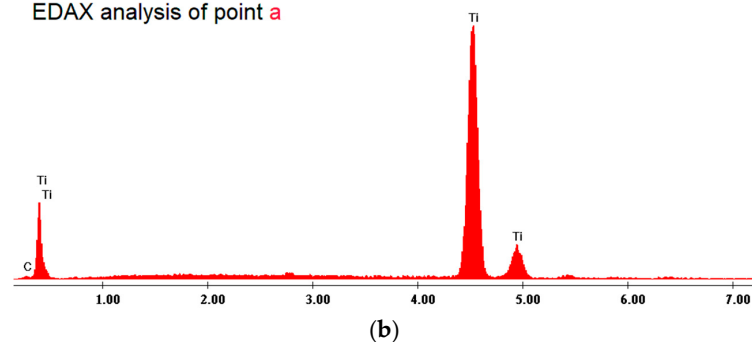


Figure 3. Cont.

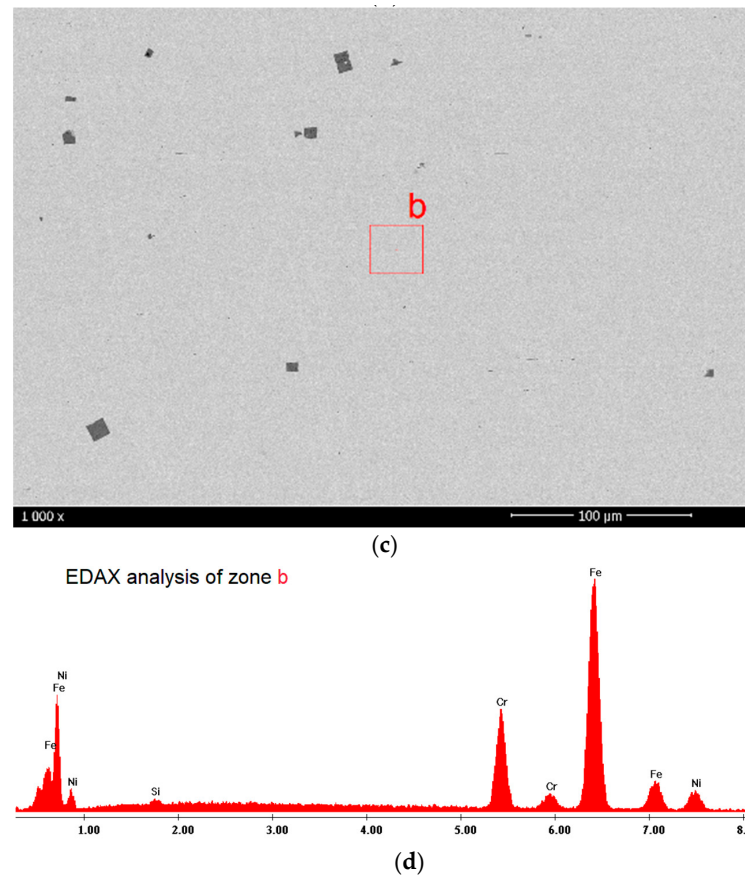


Figure 3. SEM micrographs and EDAX analysis of the SS + TiC composite coating: (a,b) TiC particles; (c,d) SS matrix.

3.2. XRD

The X-ray diffraction (XRD) technique is a highly effective analytical method to investigate the material's crystal structure, phase composition, lattice parameters and other key structural properties.

Figure 4 presents the XRD patterns for the base material unaffected by the thermal spraying process, for the powder mixture (Amperit + TiC particles) and for the surface of the deposited composite layer using the VPS method.

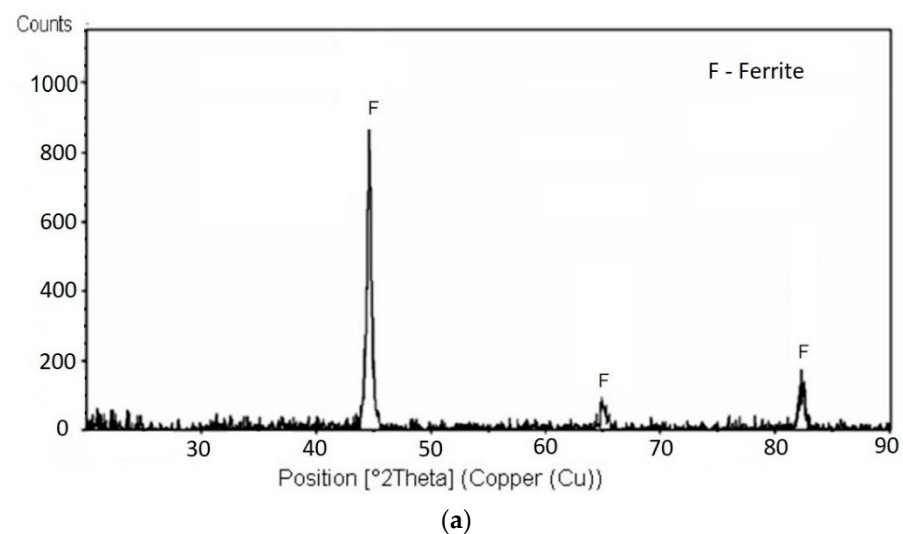


Figure 4. Cont.

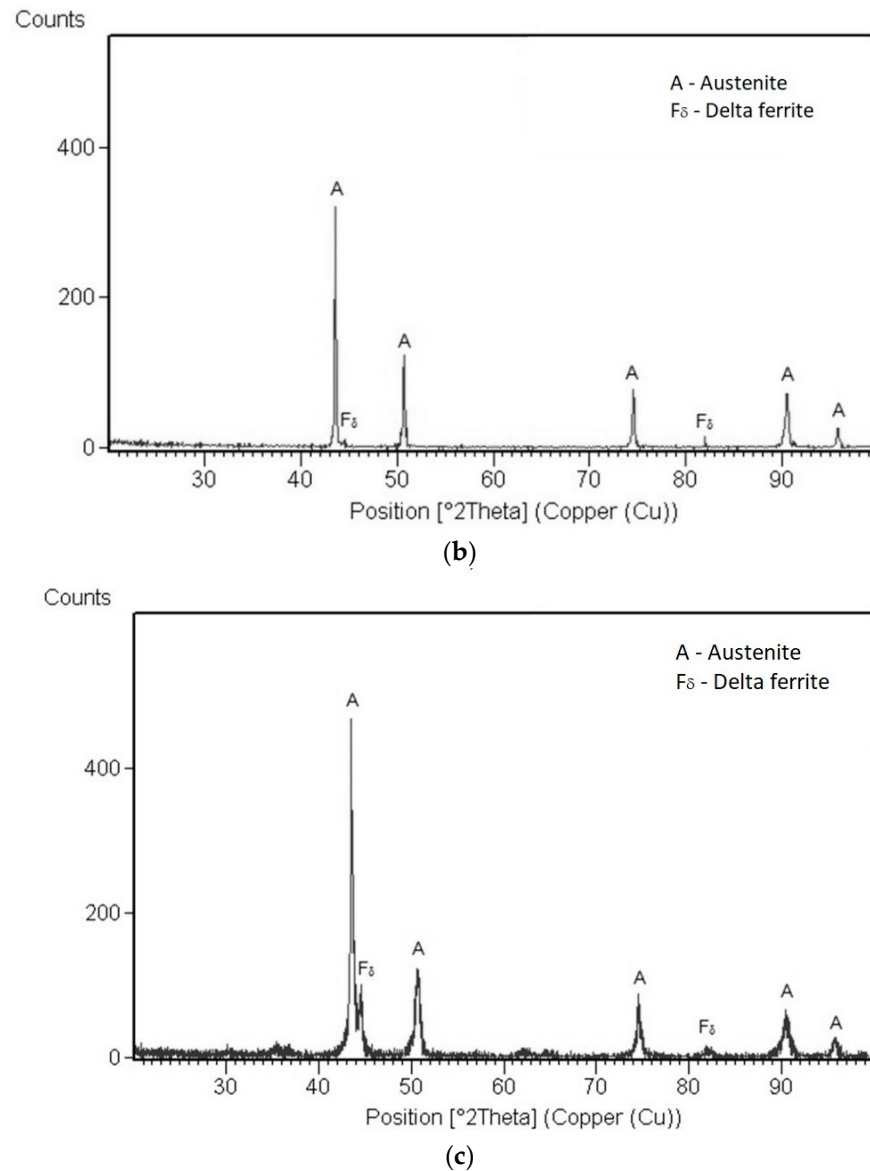


Figure 4. XRD patterns of the materials: (a) S235 base material; (b) Amperit + TiC powder mixture; (c) Amperit + TiC vacuum-sprayed coating.

By comparing the position angles of the interference peaks with the standard angles for ferrite, martensite, austenite and carbides, it is evident that, both in the base metal and in the deposited layer, the dominant phase was gamma iron (Figure 4b,c), and the TiC particles, present in small quantities, could not be detected. The diffractogram of the base metal (Figure 4a) reveals characteristic interference peaks for ferrite. These results are consistent with previous investigations conducted by other researchers [35,36].

3.3. Corrosion Behavior

The corrosion properties of the SS + TiC composite coating and of the S235 low alloyed steel were evaluated by electrochemical measurements in a 3.5% NaCl solution.

Polarization curves for each sample were drawn (Figure 5) to assess the corrosion behavior through the analysis of corrosion current intensity and electrochemical potential. An applied potential of ± 2500 mV was applied relative to the open circuit potential, with a scan speed of 1 mV/s. The measurements were made at room temperature, and by drawing the Tafel slopes, the corrosion parameters were determined (Table 3).

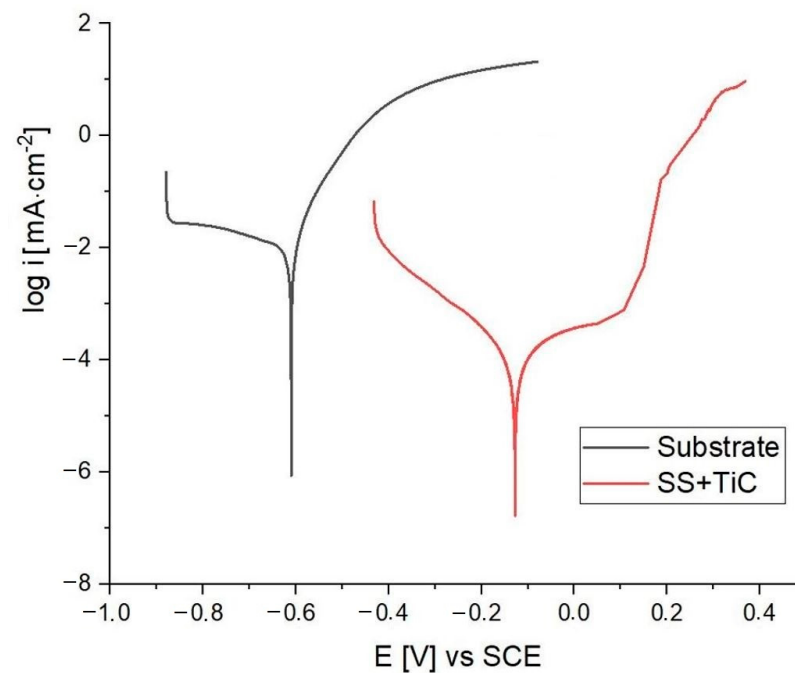


Figure 5. Polarization curves of the tested materials.

Table 3. Electrochemical corrosion data.

Sample	E [mV]	i_{corr} [$\mu\text{A}/\text{cm}^2$]
Substrate	−611.038	9.430
SS + TiC	−127.944	0.088

The area displayed by the polarization curves can be divided into two primary regions: the cathodic domain, which displays a decreasing curve and is marked by a straight Tafel line with a negative slope, and the anodic domain, which exhibits an increasing curve and is distinguished by a straight Tafel line with a positive slope.

By comparing the polarization curves of the substrate and the coating (Figure 5) and analyzing the electrochemical parameters from Table 3, one can see that the SS + TiC composite coating had much better corrosion resistance in the chloride media compared with the S235 substrate. The determined corrosion current value for the coating ($0.088 \mu\text{A}/\text{cm}^2$) was shifted to a lower value compared to the substrate ($9.430 \mu\text{A}/\text{cm}^2$), which indicates growth of the corrosion properties. It is well known that stainless steels have good corrosion properties in NaCl solutions, but one can observe that the addition of TiC particles did not decrease the chemical stability of the composite material.

3.4. Hardness and Wear Properties

Hardness and wear resistance are interconnected properties that influence the ability of a material to withstand abrasive forces, friction and other forms of mechanical stress. Higher hardness values indicate greater resistance to deformation and wear. That is why, in engineering applications, understanding the relationship between hardness and wear is crucial for selecting materials that can be exposed in specific working conditions. Improving these properties often involves selecting suitable materials, optimizing manufacturing processes and applying surface treatments or coatings to enhance performance and durability. This involves thorough material selection based on factors such as composition, microstructure and mechanical properties to ensure compatibility with the intended application. By integrating these approaches, manufacturers can effectively enhance the properties of hardness and wear resistance in materials, ensuring the reliability and longevity of components in diverse applications across various industries.

The microhardness of the composite coating and the substrate was determined in a cross section on the metallographic sample. The HV 0.3 values recorded during the measurements are displayed in Figure 6. One can observe that the coating showed higher hardness values (421 ± 8 HV 0.3) compared to the substrate (127 ± 4 HV 0.3). The variations in the hardness values for the coating are related to the distribution of TiC within the composite material. Increasing hardness was obtained because of TiC dissolution in the iron-based metallic matrix under the developed temperatures from the vacuum spraying process. The growth of the coating's hardness, along with the even distribution of carbides, strengthened the coating's resistance to deformation, allowing it to effectively disperse external stress.

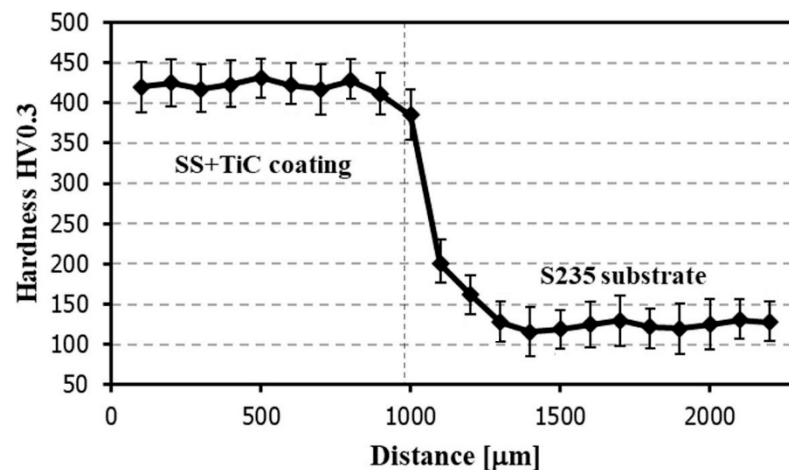


Figure 6. Hardness profile measured on the cross section of the metallographic sample.

The wear resistance of the coating and the substrate was evaluated by the pin-on-disk method. During the test, the coefficient of friction (COF) between the sample and a ball of WC was recorded. The evolution of the COF with the time is presented in Figure 7, and the obtained values are shown in Table 4. It can be seen that, during the sliding wear test, the COF for the substrate was higher ($\mu_{\text{aveg}} = 0.765$) than that recorded for the coating ($\mu_{\text{aveg}} = 0.481$). According to the friction experiments, covering the S235 substrate by vacuum spraying with the SS + TiC composite coating decreased the COF values and improved the tribological behavior of the tested material. The different values of the COF and hardness influenced the wear behavior of the tested materials (Figure 8).

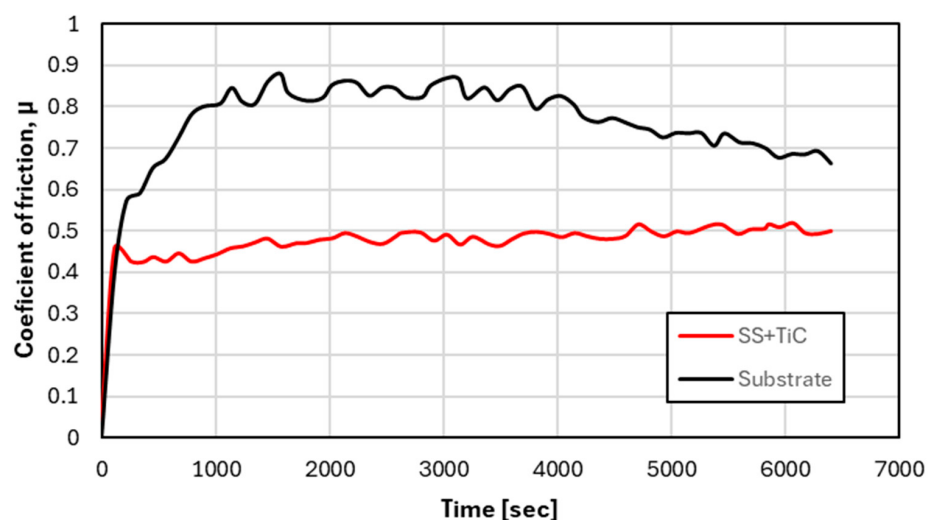
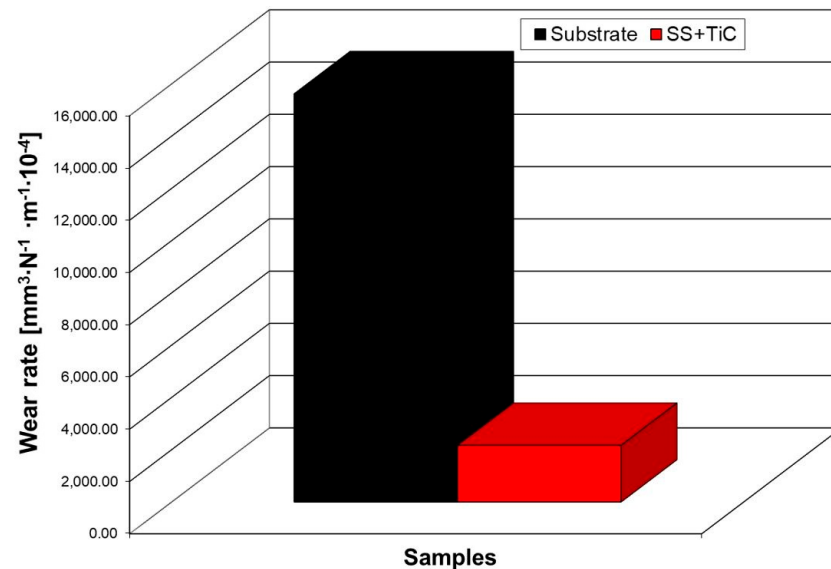


Figure 7. Coefficients of friction of the tested materials.

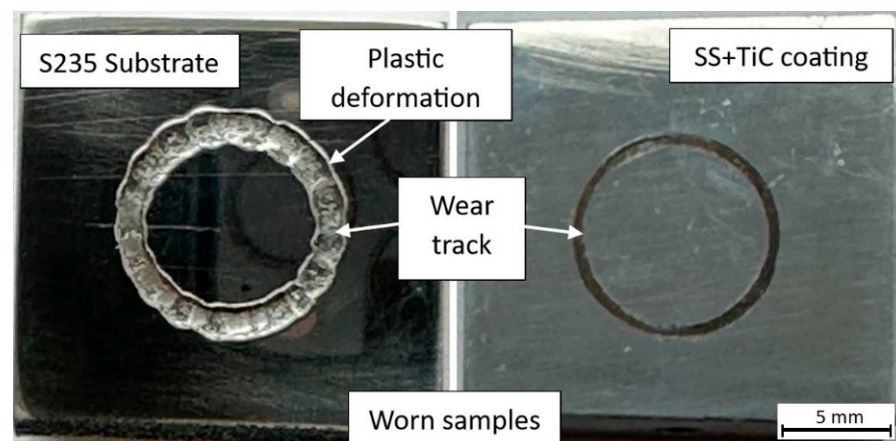
Table 4. Recorded values of the coefficient of friction (COF).

Sample	$\mu_{\min}$	μ_{aveg}	$\mu_{\max}$
Substrate	0.003	0.765	0.927
SS + TiC	0.039	0.481	0.781

**Figure 8.** Wear rate of the tested materials.

The higher hardness and lower COF values recorded for the SS + TiC composite material had a positive influence regarding the wear rates. As shown in Figure 8, the material loss decreased from $15,620 \times 10^{-4} \text{ mm}^3/\text{N}/\text{m}$ for the low alloyed steel substrate to $2167 \times 10^{-4} \text{ mm}^3/\text{N}/\text{m}$ for the iron-based composite coating, which indicates an improvement of the wear resistance of the material by approximately seven times.

The macroscopic aspect of the worn sample was in concordance with the hardness, coefficient of friction and wear rates of the tested materials. In Figure 9, one can observe that the substrate showed a wider and deeper worn track compared with the coating and, furthermore, worse wear properties. It is obvious that the wear track of the coating was shallower, and the material's degradation was uniform in comparison with the substrate material, whose worn surface suffered plastic deformation phenomena during the sliding test.

**Figure 9.** Macroscopic aspect of the wear track after the pin-on-disk test.

Based on the presented above experiments, one can say that the manufacturing of iron-based composite materials by adding TiC ceramic amounts to the chemical composition and their further spraying and deposition on the S235 substrate improved the tribological properties of the latter.

4. Conclusions

Stainless steel FeCrNiMo coatings reinforced with TiC particles were deposited by vacuum plasma spraying onto the surface of a S235 low alloyed steel substrate and were evaluated regarding their microstructure and surface properties in terms of corrosion and wear resistance.

The microstructure of the vacuum-sprayed coating appeared predominantly dense and homogeneous, with minor observable pores. Notably, due to the vacuum environment during the spraying process, no internal oxidation was identified. Furthermore, a well-defined interface between the substrate and coating was revealed, indicating favorable adherence between both system elements. Additionally, the TiC particles exhibited a uniform distribution within the stainless steel matrix. The S235 substrate showed a microstructure consisting of approximately 55%–65% ferrite, 10%–15% perlite and 20%–35% bainite.

The evaluation results of the surface properties show that both the corrosion behavior and wear properties were better in the case of the composite coating compared with the low alloyed steel substrate. The manufacturing of coatings with the addition of TiC particles dispersed in the iron-based matrix improved the hardness and sliding wear resistance of the base material. One can conclude that coating deposition using a composite material such as stainless steel reinforced with TiC can be a viable alternative to increase the corrosion and wear resistance of components that operate in such media.

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