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Microstructure and Tribological Properties of Fe-Based- Al_2O_3 - B_4C Composite Coatings Prepared by High-Velocity Arc Spraying

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Abstract: Fe-based- Al_2O_3 - B_4C coating was prepared on the low-carbon steel substrates using high-velocity arc spraying. The effects of voltage, current, and distance on the porosity and microhardness of the coating were studied by an orthogonal test, and the optimum spraying parameters were determined. The microstructure and properties of Fe-based- Al_2O_3 - B_4C coatings prepared under optimum process parameters were characterized by X-ray diffractometer (XRD), scanning electron microscopy (SEM), energy dispersive spectrometry (EDS), microhardness and friction wear tester. The results showed that the optimum process parameters were a spraying voltage of 41 V, a spraying current of 200 A, and a spraying distance of 150 mm. The porosity was $2.24 \pm 0.32\%$, and the microhardness was $1543 \pm 145 \text{ Hv}_{0.1}$, which was 8 times that of the substrate. Under the same load of 4.2 N and varying sliding speeds of 500 t/min, 750 t/min, and 1000 t/min, the coefficient of friction of the coating was less than that of the low-carbon steel, and the wear rate of the coating was 65%, 70%, and 63% lower than that of the low-carbon steel, respectively. The main wear mechanism of the coating was material spalling, accompanied by slight oxidative wear and abrasive wear.

Keywords: high-velocity arc spraying; orthogonal experiment; Fe-based composite coatings; microstructure; microhardness; tribological properties



Citation: Li, H.; Huang, F.; Lin, J.; Kang, M.; Ndumia, J.N.; Liu, J. Microstructure and Tribological Properties of Fe-Based- Al_2O_3 - B_4C Composite Coatings Prepared by High-Velocity Arc Spraying. *Coatings* **2022**, *12*, 1956. <https://doi.org/10.3390/coatings12121956>

Academic Editor: Csaba Balázs

Received: 6 November 2022

Accepted: 10 December 2022

Published: 13 December 2022

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1. Introduction

The mechanical parts of machines have served in various harsh conditions, resulting in impact vibration and wear on the surface of the material, which accelerates the failure cycle of mechanical parts. This leads to the breakdown of mechanical equipment, decreased work efficiency, and serious economic losses [1,2]. Related studies showed that thermal spraying technology could reduce surface wear of mechanical parts and improve wear resistance. Sun et al. [3] prepared a TiO_2 - Al_2O_3 composite coating on 20Mn steel using atmospheric plasma spraying (APS) technology. The hardness of the coating (66.5 HRC) increased by 220%, and the weight of wear loss was reduced by 86.2% compared to 20Mn steel. Zhang et al. [4] fabricated Fe-based amorphous coatings on AISI 1045 mild steel using high-velocity oxygen fuel (HVOF). The Fe-based amorphous coating has a higher hardness, about six times that of the substrate. At the same load and sliding speed, the Fe-based amorphous coating exhibited a more stable coefficient of friction (COF) and a lower wear rate than the substrate. Ma et al. [5] fabricated a WC-10Co-4Cr coating on 65Mn steel by supersonic flame spraying. The results showed that the average microhardness of the WC-10Co-4Cr coating increased by 143%, and the wear weight loss decreased by 53% compared with the traditional heat treatment (quenching + medium temperature tempering) of 65Mn steel. Among thermal spraying technologies, high-velocity arc spraying (HVAS) is widely used in the reinforcement, repairing, and remanufacturing of mechanical parts because of its stable spraying quality, high spraying efficiency, low energy consumption, and cost.

At present, the main coatings used for HVAS include Fe-based, Ni-based, and Co-based coatings. Among them, Ni-based and Co-based coatings have excellent self-solubility, mechanical properties, and high-temperature thermal stability. However, Ni-based and Co-based

are relatively more expensive and difficult to achieve wide application on the surface of mechanical parts. Fe-based coatings are widely used in HVAS due to their high toughness, special amorphous forming ability, lower production costs, and availability of raw materials. These properties make them suitable replacements for Ni-based and Co-based coatings in ambient and high-temperature applications [6,7]. Tian et al. [8] prepared FeNiCrAl coating on 42CrMoA. The coating had high hardness (676 Hv_{0.3}), and the two oxide films of Fe₃Al and Al₂O₃ that were generated on the worn surface during friction-wear improved the wear resistance of the coating. Hard ceramic particles, such as Cr₃C₂, Al₂O₃, B₄C, TiB₂, etc., are often added to the cored wire to form a metal/ceramic composite cored wire, which combines the mechanical properties and toughness of metal materials with high hardness, excellent tribological properties and high-temperature oxidation resistance [9,10]. Yasir et al. [11] prepared Fe-based-Al₂O₃ composite coatings on AISI 1045 steel by HVOF. The Al₂O₃ particles were uniformly distributed in the coating. Compared with the amorphous coating, the composite coating showed higher wear resistance and lower COF in both lubricated and dry conditions. Yoon et al. [12] prepared Fe-based metal bulk metallic glass (BMG)-B₄C composite coatings on low-carbon steel by APS. Adding B₄C particles reduced the COF and wear weight loss of the composite coatings because the B₄C particles played the role of a solid lubricant in sliding friction wear tests, increased the hardness of the wear surface, and inhibited the extension of plastic flow. Datye et al. [13] used APS to prepare Al₂O₃-B₄C composite coatings on plain-carbon steel. It was found that no chemical reaction occurred between the Al₂O₃ and B₄C particles, retaining their initial phases before spraying. Compared with the Al₂O₃ coating, the thermal and mechanical properties of the coating with B₄C particles were improved. Cao et al. [14] prepared NiCoCrAlY-Al₂O₃-B₄C composite coating on 45 steel by APS. The coating had less wear debris and scratches after wear, a smaller COF (0.46) at room temperature, and better high-temperature oxidation resistance.

The above studies showed that the composite coatings with Al₂O₃-B₄C particles exhibit high hardness and good wear resistance. However, most researchers prepared Al₂O₃-B₄C composite coatings by APS. In the previous research, our group prepared FeMnCrNi coating on low-carbon steel by HVAS, but the coating has low hardness (353.4 Hv_{0.1}) and poor wear resistance [15]. In order to improve the hardness and wear resistance of the coating, Al₂O₃ and B₄C particles were added to the Fe-based cored wire. The effects of spraying voltage, spraying current, and spraying distance on the porosity and microhardness of the coating were studied by orthogonal test. The composition, microstructure, and mechanical and tribological properties of the coating under the optimum spraying process parameters were studied. This study is expected to provide a reference for the restoration, enhancement, and remanufacturing of mechanical parts.

2. Materials and Methods

2.1. Materials

The coating material used for this test was Fe-based-Al₂O₃-B₄C cored wire. The self-designed cored wire had a diameter of 2 mm. Particle size was 30–50 µm, a filling rate of 30%, and the outer sheath material was 308 stainless steel. The substrate material was low-carbon steel with a size of 50 mm × 30 mm × 8 mm. Tables 1 and 2 show the composition and content of the cored wire and low-carbon steel, respectively.

Table 1. Chemical composition of Fe-based-Al₂O₃-B₄C cored wire (wt. %).

Cored Wire	Fe	Mn	Cr	Ni	Al ₂ O ₃	B ₄ C
Fe-based-Al ₂ O ₃ -B ₄ C	Bal.	10–15	8–15	≤5	10–14	15–21

Table 2. Chemical composition of low-carbon steel (wt. %).

Substrate	Fe	C	Si	Mn	P	S
low-carbon steel	Bal.	0.14–0.22	≤0.30	0.30–0.65	≤0.045	≤0.050

2.2. Methods

2.2.1. Preparation of Coatings

Firstly, the substrate was polished before spraying and ultrasonically cleaned with an acetone solution to remove surface contaminants. Secondly, the substrate was roughened by sandblasting with 16 mesh iron sand, the air pressure was 0.6 MPa, and the angle of sandblasting was 70–90°. Finally, the Fe-based- Al_2O_3 - B_4C coating was prepared on the low-carbon steel surface using arc spray equipment (LSARC500S, Shanghai Liangshi Intelligent Technology Co. Ltd., Shanghai, China). In this experiment, spraying voltage, current, and distance were used as the process variables for optimization. Porosity and hardness were used as the response values. According to the orthogonal experimental method, three factors and three levels of the $L_9 (3^4)$ orthogonal test table were designed. Table 3 shows the factors and levels of the orthogonal test.

Table 3. Factors and levels of orthogonal test.

Levels	Factors		
	A Spraying Voltage/V	B Spraying Current/A	C Spraying Distance/mm
1	33	160	150
2	37	180	200
3	41	200	250

2.2.2. Characterization

The phase composition of the coating was analyzed by X-ray diffractometer (XRD, PANalytical B.V., Almelo City, The Netherlands) with the following parameters: emission target used in the tests was Cu-K α radiation, the wavelength $\lambda = 0.154056$ nm, the accelerating voltage 40 kV, current 40 mA, the scanning rate 2°/min and the scanning angle 10–90°.

The surface morphology, cross-sectional morphology, and wear morphology of the coating were observed by the scanning electron microscope (SEM, Quanta FEG 250, Waltham, MA, USA). The accelerating voltage was 20 kV, and the magnification ranged from 200 $\times$ to 1000 $\times$. The distribution of chemical elements in the coatings was analyzed by an energy-dispersive spectrometer (EDS, Bruker, Bremen, Germany).

The porosity of the coating was determined by the greyscale stretching method using ImageJ software [16]. Fifteen areas were randomly selected on the cross-section of the coating, and the results were averaged.

The microhardness of the coating was measured by a microhardness tester (Duramin-40 A1, Struers, Ballerup, Copenhagen, Denmark) [17] with a load of 100 gf, or a loading time of 15 s. A total of 15 points were measured, and the average was taken to determine the microhardness. The vertical distance between two adjacent points was 80 μm .

The friction and wear properties of the coatings were tested by a comprehensive material performance surface tester (CFT-I, Lanzhou Zhongke Kaihua Technology Development Co., Lanzhou, China). Before the test, the samples were ground and polished to maintain the same surface roughness ($R_a = 0.8$ μm) according to ASTM G133–05 [18]. The counterpart was a Si_3N_4 ($R_a = 0.1$ μm) ceramic ball with a diameter of 4 mm. The test conditions and parameters were as follows: room temperature without lubrication, sliding speeds of 500 t/min, 750 t/min, 1000 t/min, applied load of 4.2 N, sliding time of 30 min, and reciprocating stroke of 2 mm. The width, depth, and three-dimensional morphology of the wear scar were observed by a Laser confocal microscope (Olympus Corporation, OLS 4100, Tokyo, Japan). Further analysis of the surface morphology after wear was done by SEM to analyze the wear mechanism. The volume wear rate (W_r) was used to characterize the wear resistance of the coating and is given by Equation (1) [19]:

$$W_r = \frac{S_s \times L_1}{F \times L_2} \quad (1)$$

where S_s is the cross-sectional area of the wear scar (mm^2), L_1 is the reciprocating length (mm), F is the normal load (N), and L_2 is the sliding distance (m).

3. Results and Discussion

3.1. Results of the Orthogonal Test

Table 4 shows the results of the orthogonal tests. In practical analysis, the quality of the coating is evaluated by numerous indicators, among which hardness represents the coating's ability to resist deformation and significantly influences the wear resistance of the coating [20,21]. As the weakest point of the coating, the pores become the source of cracks and corrosion during the service life of the coating [22,23]. In this experiment, microhardness and porosity were used as evaluation indicators of coating quality to obtain the optimal process parameters. In order to unify the influence of microhardness and porosity on coating performance, the microhardness and porosity were weighted and rated, and multiple indicators were converted into one comprehensive indicator. The comprehensive rating was set to 100 points, of which the hardness was 60 points, and the porosity was 40 points. The comprehensive weighted rating Y_i was calculated using the formula [24]:

$$Y_i = a_{i1}y_{i1} + a_{i2}y_{i2} + \dots + a_{ij}y_{ij} \quad (2)$$

where a_{ij} is the coefficient, y_{ij} is the test indicator, and i, j represent the j th indicator of the group i test.

Table 4. Orthogonal test results.

No.	A Spraying Voltage /V	B Spraying Current /A	C Spraying Distance /mm	Porosity /%	Microhardness/Hv _{0.1}	Comprehensive Rating
1	33	160	150	2.60	1152.58	85.50
2	33	180	200	3.33	1104.82	53.80
3	33	200	250	2.36	1182.37	98.04
4	37	160	200	2.28	1363.89	127.95
5	37	180	250	3.27	1337.89	90.78
6	37	200	150	2.82	1478.70	127.02
7	41	160	250	3.07	1427.16	110.89
8	41	180	150	2.14	1490.76	151.69
9	41	200	200	2.90	1494.01	126.63

The range of variation for each indicator is K :

$$K_1 = 3.33 - 2.14 = 1.19 \quad (3)$$

$$K_2 = 1494.01 - 1104.82 = 389.19 \quad (4)$$

where K_1 is the range of porosity and K_2 is the range of hardness.

The porosity was given a negative value because it belongs to the smaller-the-better characteristic, and microhardness was given a positive value because it belongs to the larger-the-better characteristic. The porosity coefficient a_{i1} and the microhardness coefficient a_{i2} are given by:

$$a_{i1} = -\frac{40}{K_1} = -33.6 \quad (5)$$

$$a_{i2} = \frac{60}{K_2} = 0.15 \quad (6)$$

From the above equations, the comprehensive performance rating Y_i of each test group was calculated as follows:

$$Y_i = -33.6Y_{i1} + 0.15Y_{i2} \quad (7)$$

3.2. Analysis of Test Results

The test uses range analysis to analyze the effect of each factor on the quality of the coating. The results of range analysis for orthogonal experiments are shown in Table 5. Level 1, Level 2, and Level 3 in the table represent the average values of each factor at each level, respectively. The larger the average value, the better the indicator of coating performance. R represents the range between the average values corresponding to each level of each factor, reflecting the importance of the factor. The larger R indicates that the factor has a greater influence on the output value. According to the level value of the three factors of the orthogonal test, Figure 1 shows the three factors on the x-axis and the sum of the average value of the three parameters under the same factor on the y-axis.

Table 5. Range analysis results of Orthogonal tests.

Factors	Porosity/%				Microhardness/Hv _{0.1}				Comprehensive Rating			
	Level 1	Level 2	Level 3	R	Level 1	Level 2	Level 3	R	Level 1	Level 2	Level 3	R
A Spraying voltage	2.76	2.79	2.70	0.09	1146.59	1393.49	1470.64	324.05	79.11	115.25	129.74	50.63
B Spraying current	2.65	2.91	2.84	0.26	1314.54	1311.16	1385.03	73.87	108.11	98.76	117.23	18.47
C Spraying distance	2.52	2.84	2.90	0.38	1374.01	1320.91	1315.81	58.20	121.40	102.79	99.90	21.50
Major-minor order	C > B > A				A > B > C				A > C > B			
Best plan	A3B1C1				A3B3C1				A3B3C1			

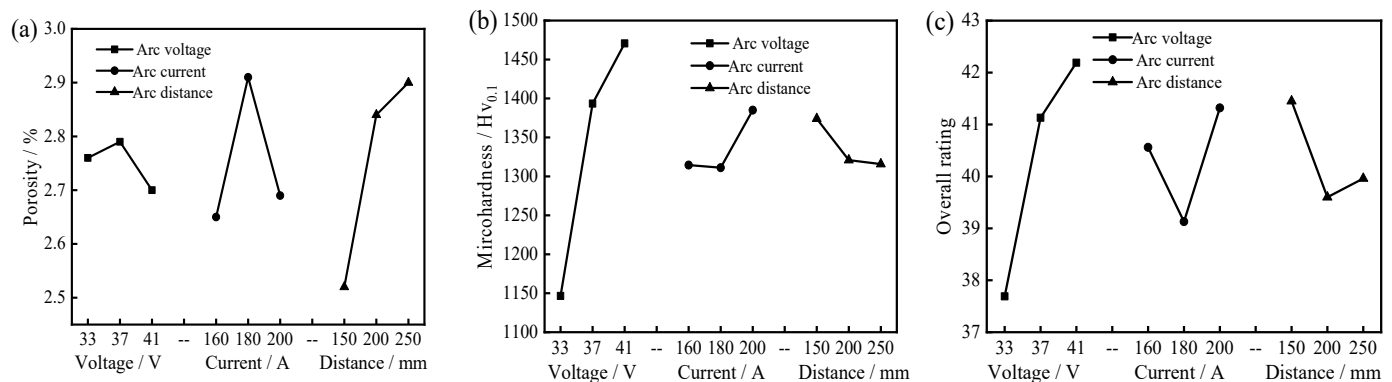


Figure 1. Horizontal trend diagram of orthogonal test with different target parameters (a) porosity; (b) microhardness; (c) comprehensive rating.

Combined with Table 5 and Figure 1, the order of the variables affecting the comprehensive performance of the coating are as follows: spraying voltage, spraying distance, and spraying current. The comprehensive performance of the coating improved with the increase in spraying voltage. At low spraying voltage, the spraying power is low, resulting in the incomplete melting of particles. Thus, the particles will be insufficiently deformed when they reach the substrate surface. The splat particles have a weak mechanical bond resulting in higher porosity and lower microhardness of the coating. As the spraying voltage increases, the enthalpy and velocity of the particles increase, decreasing the number of unmelted particles. This promotes the fusion of the molten droplets and the ceramic phase, reduces the gap between the layers, and increases the density and hardness of the coating [25]. Kang et al. [26] also suggested that the increased spraying voltage increases the melting rate of droplets, which increases the denseness of the coating. With the increase in spraying current, the comprehensive performance of the coating first decreased and then increased. The analysis suggests that the spraying current increased the temperature of the molten droplets so that the molten droplets still maintain a high temperature when they reach the substrate surface and have good fluidity, which is beneficial to fill the pores

between the coatings. The variation of pores with the spraying current was consistent with that obtained by Zhou et al. [27]. With the increase in the spraying distance, the flight time of the droplets in the air increases. Subsequently, the higher oxide content in the coating increases due to the oxidation of the molten droplets during the flight process. The increase in spraying distance also leads to the decrease of enthalpy and fluidity of molten droplets, thus increasing the porosity of the coating and reducing the comprehensive performance of the coating [28,29]. Mauer et al. [30] found that the increased spray distance reduces the droplet temperature and increases the porosity of the coating. When the spraying current and distance were 200 A and 250 mm, respectively, the temperature of the molten arc area increased and formed tiny droplets. However, the flight time of the droplets in the air increased, leading to a lower temperature of the molten droplets before reaching the substrate. This reduced the spreadability of the droplets hence, increasing the internal defects of the coating. Therefore, the comprehensive performance of the coating was best when the spraying voltage, current, and distance were 41 V, 200 A, and 150 mm, respectively.

The Fe-based- Al_2O_3 - B_4C coating was fabricated using the optimal spraying process parameters. The microhardness of the coating was $1543 \pm 145 \text{ Hv}_{0.1}$, and the porosity was $2.24 \pm 0.21\%$. After being substituted in Equation (7), the comprehensive rating was 156.16. Compared with Table 4, the coating fabricated using the optimal parameters has a higher comprehensive rating.

3.3. X-ray Diffraction Analysis of Coating

Figure 2a shows the X-ray diffraction pattern of the raw material powder. It can be seen that the metal and ceramic particles in the powder core exist in elemental form. Figure 2b shows the XRD diffraction pattern of the coating after optimizing the parameters. The physical phases of the coating after spraying include the following phases: α -Fe (PDF#06-0696) phase, (Fe, Cr) (PDF#41-1466) solid solution phase, Fe-Cr (PDF#34-0396) phase, Al_2O_3 (PDF#04-0877) phase, B_4C (PDF#35-0798) phase and Fe_3B (PDF#34-1198) phase. Since the (Fe-Cr) solid solution has an FCC lattice structure similar to that of α -Fe, there are no pure Cr diffraction peaks after spraying, and the radius of Fe and Cr is close to each other with good mutual solubility so that the Cr element dissolves in the Fe element [31,32]. At diffraction angles of $2\theta = 44.78^\circ$, more diffuse diffraction peaks appear, indicating that the coating contains a small amount of amorphous content. Inoue and Molokanov et al. [33,34] believed that the large atomic radius differences would form a close and disordered stacking between atoms. This structure can increase the viscosity of the alloy in the supercooled liquid region, thus hindering the long-range diffusion of atoms and inhibiting the nucleation and growth of crystals. It can be seen from Tables 6 and 7 that B and C atoms form a large atomic radius difference from other metal elements, and the addition of B and C also makes the alloy elements have a large negative mixing enthalpy, which also has the formation of an amorphous phase. Additionally, the high-velocity air pressure of the HVAS process provided a high cooling rate that resulted in the internal atoms of molten particles being cooled into short-range ordered and long-range disordered amorphous structures before being arranged into regular ordered crystals [12,35,36].

Table 6. Atomic radii of different elements.

Element	Fe	Mn	Cr	Ni	Al	O	B	C
Atomic radius/nm	0.124	0.127	0.125	0.125	0.143	0.074	0.082	0.077

Table 7. Mixing enthalpy values of atomic pairs.

Components of Alloy	Fe-B	Fe-C	C-Cr	Ni-B	Cr-B	Mn-C	Fe-Mn	Fe-Cr	Fe-Ni
Mixing enthalpy values/(kJ·mol ^{−1})	−26	−50	−61	−24	−31	−66	0	−1	−2

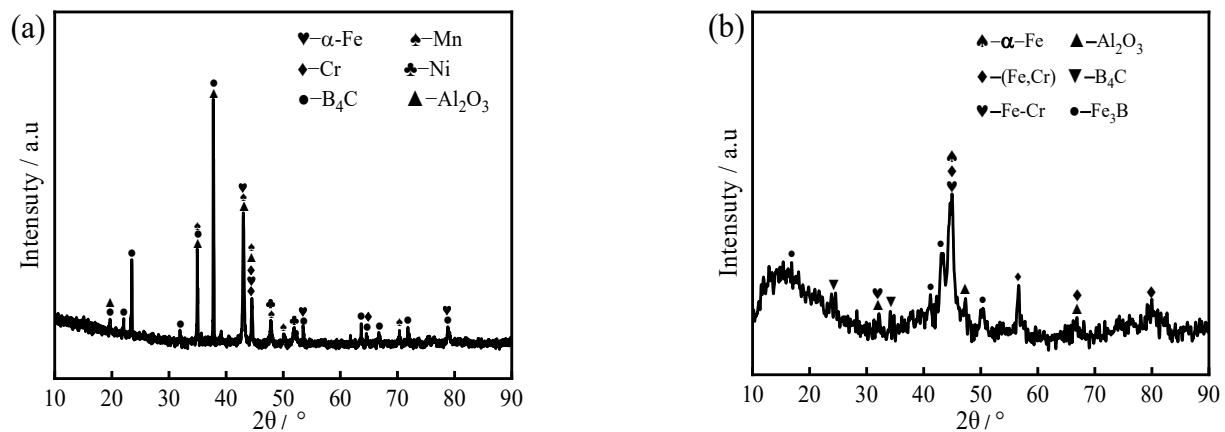


Figure 2. XRD pattern (a) powder; (b) Fe-based- Al_2O_3 - B_4C coating.

3.4. Surface and Cross-Sectional Morphology of the Coating

Figure 3 shows the surface microstructure of the coating. The surface is irregular, with pores, micro-cracks, and un-melted particles. Molten droplets spread in a disc shape on the coating surface. During the spraying process, the cored wires were heated to form molten or semi-molten particles. Some of the particles with a higher melting point were unmelted and insufficiently deformed when they hit the substrate surface under the action of high-velocity air pressure. Thus, particles were embedded in their original form in the coating surface during the solidification and stacking process [25,37,38]. At the same time, a faster cooling rate caused the droplets to shrink, limiting the droplets' tendency to spread. Internal stresses were generated during the deposition process, leading to micro-cracks on the surface of the coating [39].

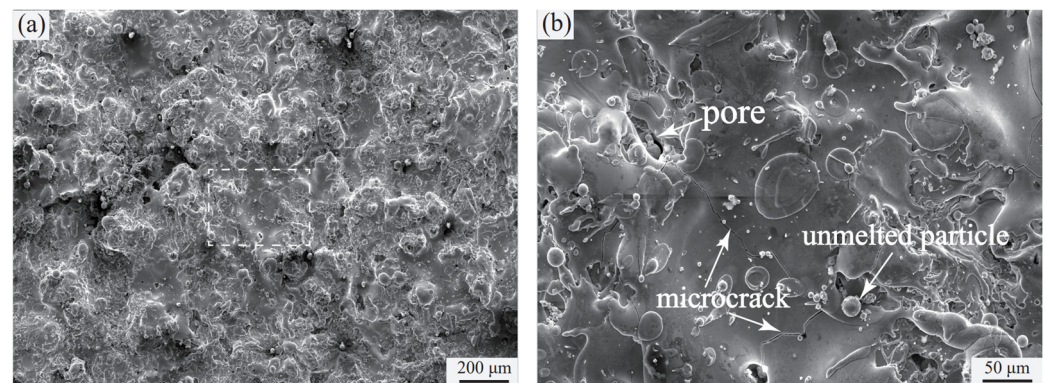


Figure 3. Surface morphology of coating (a) magnification of 200 $\times$; (b) magnification of 500 $\times$.

Figure 4 shows the cross-section morphology of the coating. The average thickness of the coating is $451 \pm 16 \mu\text{m}$, with a typical layered structure. The cross-section contains some pits, microcracks, and unmelted particles. The cross-section images have some pits that formed when the internal hard phases and un-melted particles fell off during the polishing and grinding of the cross-sectional samples. In addition, irregularly shaped pores and micro-cracks can be observed at the interlayer boundaries due to the incomplete stacking of droplets and the untimely removal of gas during droplet deposition [23]. Table 8 summarizes the EDS results of the light grey area I, the dark grey area II and the black area III. The main composition of the light grey area I is 76.85% Fe, 14.77% Cr and a small amount of Mn, Ni, and Al elements. The light grey area I is mainly composed of Fe-Cr metal compounds, which is consistent with the XRD results.

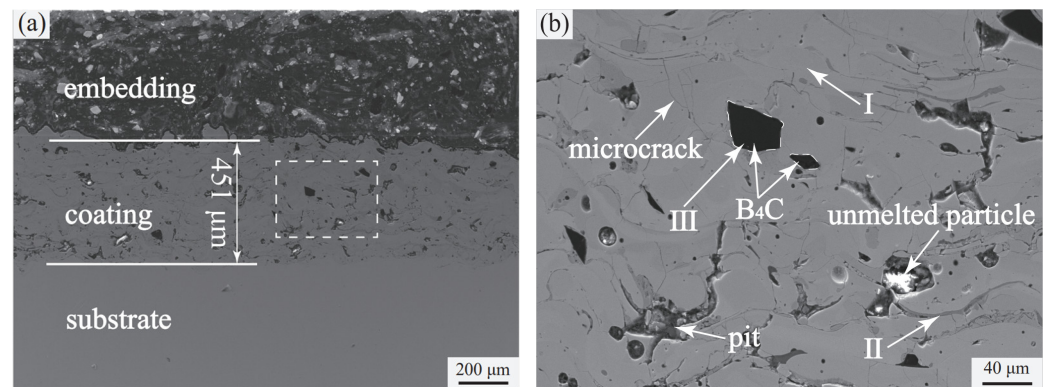


Figure 4. Cross-sectional morphology of coating (a) at 200 $\times$; (b) at 1000 $\times$.

Table 8. The results of EDS in different areas (wt. %).

No.	Main Element Content								
	Fe	Cr	Mn	Al	Ni	O	C	B	Si
I	76.85	14.77	6.57	0.02	1.80	-	-	-	-
II	30.06	6.13	27.78	0.22	35.80	-	-	-	-
III	-	-	-	0.19	-	0.19	19.47	77.38	2.77

Figure 5 shows the EDS line scan of the coating-substrate interface. The interface is an uneven curve without obvious pores, cracks, and other defects. This can be attributed to the surface roughness of the substrate achieved by sandblasting. The molten droplets collided, deformed, and diffused into flakes, and the uneven surface of the substrate interlocked to form an uneven curve. Combined with EDS line scanning, the elemental content of Fe, Mn, Cr, and Ni suddenly decreased while the elemental content of Al and O suddenly increased after passing through area A. This indicates that the Al_2O_3 particles are embedded inside the coating, which conforms to the XRD results.

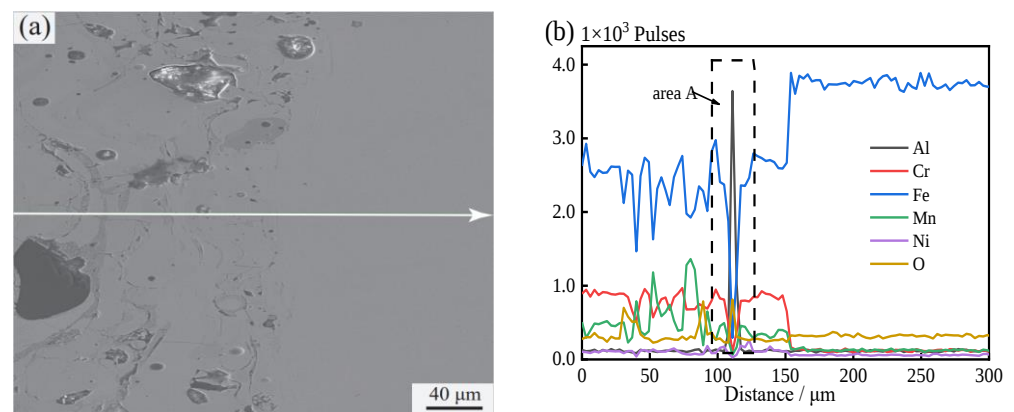


Figure 5. EDS line scan analyses of the interfaces between coating and substrate (a) SEM cross-sectional image; (b) Line scan profile for the coating-substrate interface.

Figure 6 shows the schematic diagram of the deposition process of the coating. The sprayed particles are composed of metal droplets and ceramic particles. The coating was formed by co-deposition in the solid-liquid two-phase state. This was due to the high agglomeration strength of ceramic particles, which can only rupture during the spraying process and are difficult to deform plastically. Thus, hindering the mutual diffusion between the coating and the substrate and reducing the tensile bond strength between the coating and the substrate [40]. The interface has a transition layer of 37 μm , so the main combination was mechanical bonding between the coating and the substrate. Qin et al. [41]

also confirmed that ceramic particles hinder inter-diffusion between the coating and the substrate, forming a mechanical bond.

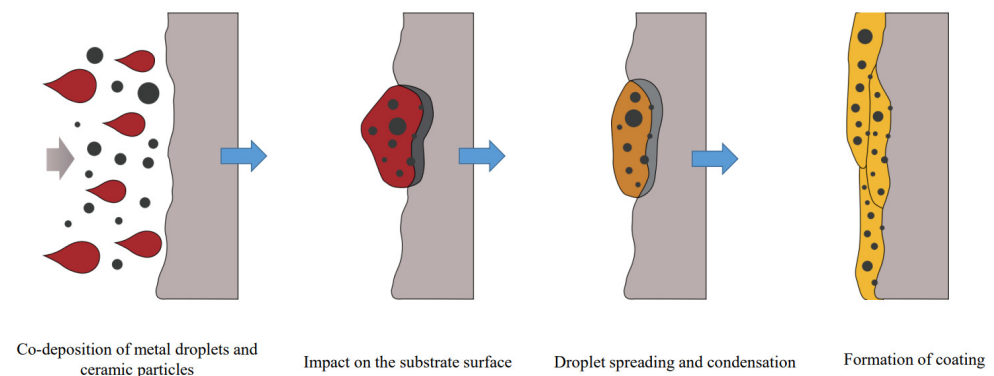


Figure 6. Schematic diagram of the deposition process of the coating.

3.5. Coating Microhardness

Figure 7 shows the microhardness distribution curve of the coating along the cross-section. The coating hardness fluctuates considerably, from 1431 Hv_{0.1} to 1730 Hv_{0.1}, mainly due to the aggregation and uneven distribution of Al₂O₃, B₄C, and Fe₃B hard phase. The average microhardness of the coating is 1543 ± 145 Hv_{0.1}, which is eight times higher than that of the low-carbon steel. Combined with XRD and EDS analysis, there are three reasons for the relatively high hardness of the coating. Firstly, the (Fe, Cr) solid solution resulted in lattice distortion, thereby preventing the movement of crystal boundaries and dislocations, and played a solid solution strengthening effect [42,43]. Secondly, hard phase particles of Al₂O₃, B₄C, and Fe₃B were dispersed in the coating, which plays a dispersion-strengthening effect. Thirdly, the small amount of amorphous phase structure in the coating reduces crystal defects of dislocation, stacking, and composition segregation, which improves the resistance of the coating to deformation [35]. Figure 8a shows the indentation of the hardness of the low-carbon steel. It can be seen that there was obvious plastic deformation at the indentation, attributed to the low hardness and strong plasticity of the low-carbon steel. Figure 8b shows the hardness indentation of the coating cross-section, where the crack extensions appear around the diamond-shaped indentation. The hard phases, such as Al₂O₃, B₄C, and Fe₃B, increased the brittleness of the coating. This is consistent with the conclusions of Motas et al. [44].

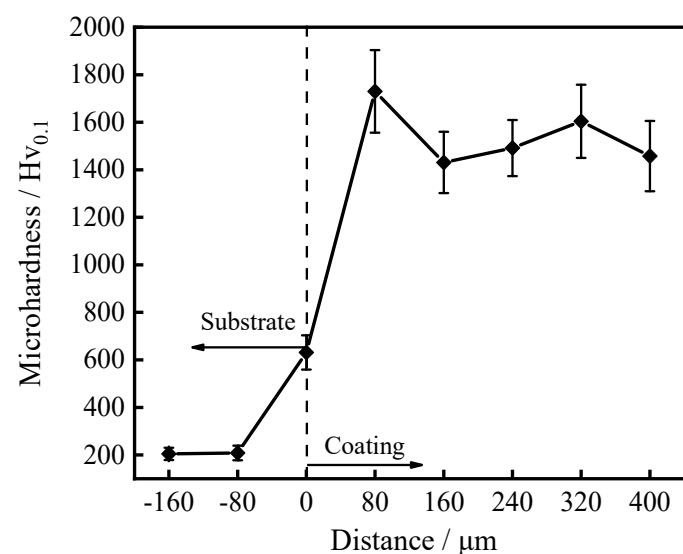


Figure 7. Cross-section hardness distribution curve.

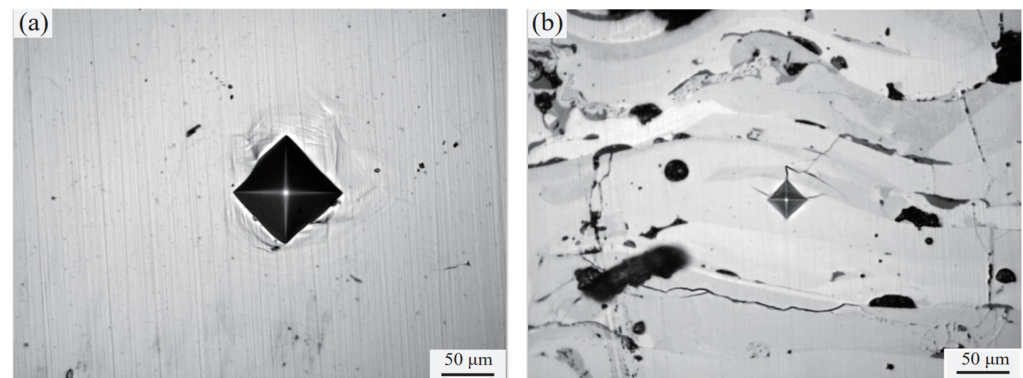


Figure 8. Cross-section hardness indentation (a) low-carbon steel; (b) Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating.

3.6. Wear Resistance

Figure 9a,b shows the COF of the low-carbon steel and coating at different sliding speeds. The COF of low-carbon steel is greater than the COF of the coating at different speeds under the same load. Under the reciprocating action of the load, the hard reinforcement phases play the role of a lubricant in the friction process, reducing the COF. The high hardness of the coating increased the resistance to plastic deformation and enhanced the resistance to cutting and ploughing, resulting in a lower COF of the coating [45]. However, the COF of the coating was unstable and fluctuated more than that of the substrate. The irregular coating structure with dispersed hard phases and spalling pits caused an uneven wear surface. Under the action of reciprocating normal and tangential forces, the hard phases in the coating fell off. Extrusion deformation and shear failure occurred within the spalling pits, making the contact of the counterpart fluctuate greatly.

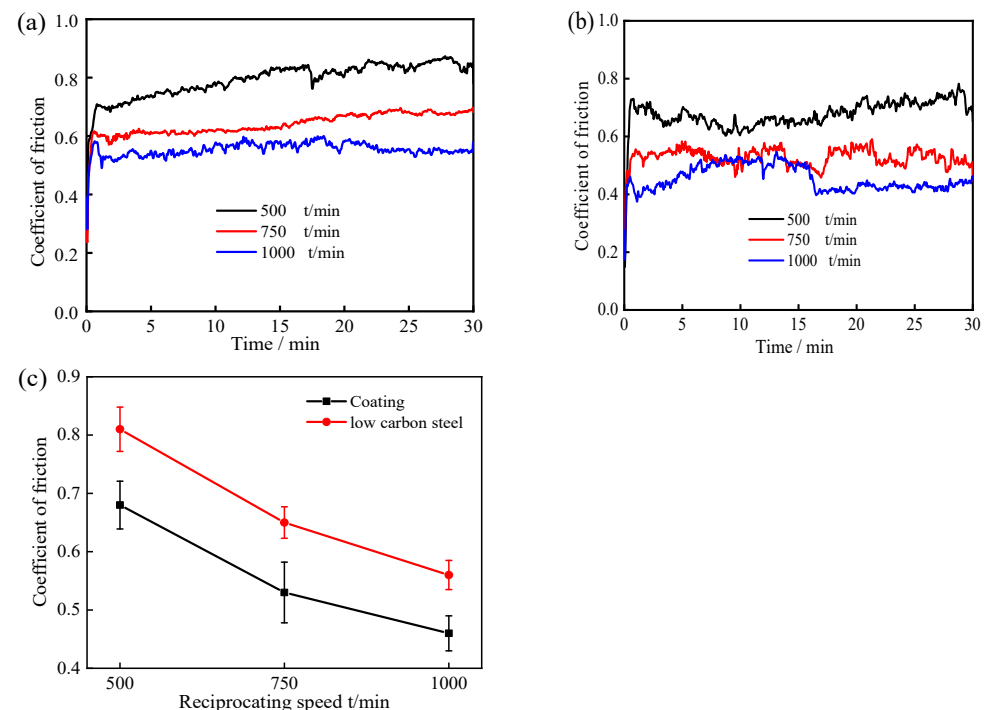


Figure 9. COF of the low-carbon steel and coatings at sliding speeds of 500 t/min, 750 t/min, and 1000 t/min (a) low-carbon steel; (b) Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating; (c) Average COF.

Sliding speed is an important factor influencing the wear mechanism, impacting the material, causing mechanical deformation, and changing the friction interface's temperature [46]. Figure 9c shows the trend of the average COF of the substrate and coating at different sliding speeds. The COF of the substrate and coating decreases with the increase

in sliding speed. Due to the low sliding speed, friction mainly manifests as local adhesion and shear, causing a local temperature increase, but this thermal effect does not change the wear mechanism. As the sliding speed increased, the number of frictional contacts at the same point increased and shortened the heat dissipation time. The surface oxidation increased, and the mechanical properties of the coating, such as hardness and shear strength, decreased. As a result, the high temperature changed the properties of the friction material, reducing the sliding resistance and the COF. Tillmann et al. [47,48] suggested that the decrease in the COF can be attributed to the production of oxides caused by repeated friction, which act as solid lubricants.

Figure 10a–c shows two-dimensional profile curves of the wear scar of the substrate and coating at different sliding speeds. The wear scar width and depth of the low-carbon steel and the coating increased with the increase in the sliding speeds. The two-dimensional wear scar curve of the coating at different sliding speeds was smaller than that of the low-carbon steel. The higher hardness of the coating increases its resistance to plastic deformation. The (Fe, Cr) solid solution phase and Al_2O_3 , B_4C , and Fe_3B hard phases contribute to the solid solution strengthening and dispersion strengthening effect that improves the bearing capacity of the coating [14]. Figure 10d shows the trend of low-carbon steel and coating wear rates at different sliding speeds. Under the same load, the wear rate of the low-carbon steel and the coating increased with the increase in the sliding speed. The wear rates of the Fe-based- Al_2O_3 - B_4C coating at 500 t/min, 750 t/min, and 1000 t/min are 65%, 70%, and 63% lower than that of the low-carbon steel, respectively. At lower sliding speeds, the wear rate of the coating increased slowly, while at higher speeds, the wear rate increased rapidly. This is because, at lower sliding speeds, the surface temperature gradually increases, forming an oxide film on the worn surface that decelerates the wear rate. Vashishtha et al. [49] believed that the increase in sliding speed increases the friction temperature, produces oxides, and reduces the COF and wear rate. However, if the sliding speed is too high, it causes the oxide film in the wear area to break, and the fine abrasive particles produce micro-cutting of the coating, which aggravates the wear rate of the coating.

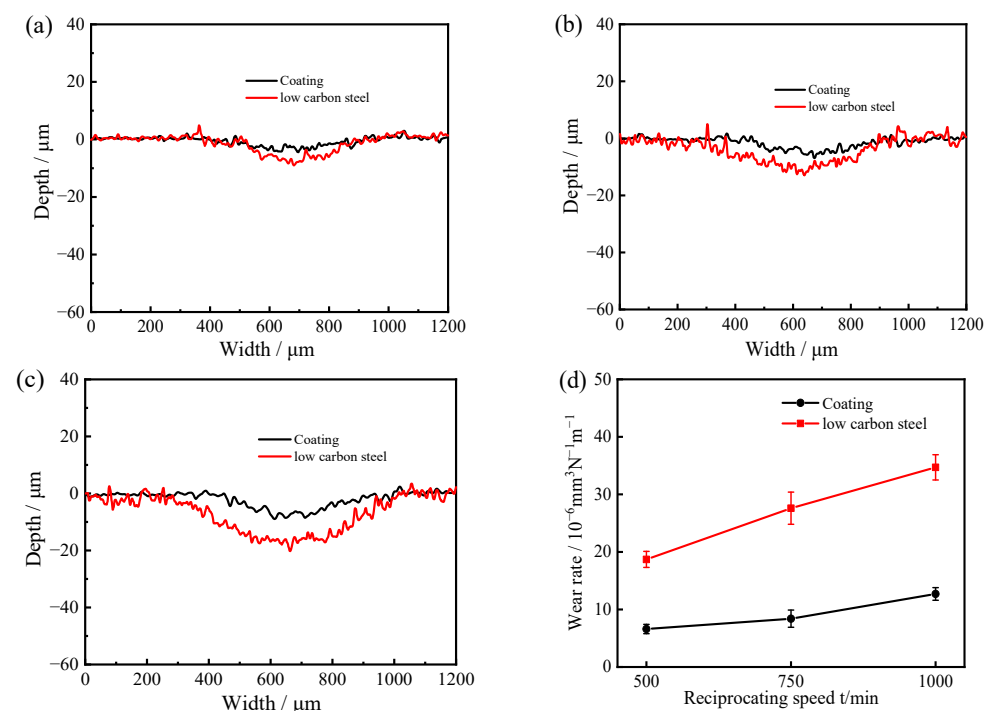


Figure 10. The profiles of the wear section and wear rates of the low-carbon steel and coatings at different sliding speeds (a) 500 t/min; (b) 750 t/min; (c) 1000 t/min; (d) Wear rates.

In order to study the wear mechanism, the SEM images of the worn surfaces were analyzed. Figure 11a–d shows the morphology of the wear scars on the substrate at sliding speeds of 500 t/min and 1000 t/min. A large area of spalling pits and deep grooves appeared on the substrate as the sliding velocity increased. This is due to the low hardness and high plasticity of low-carbon steel. Under the repeated action of high sliding speed, the temperature of the friction surface rose, and the contact point softened, resulting in a gradual weakening of the surface resistance to plastic deformation. The peeled-off debris caused the worn surface to form spalling pits and grooves. It has also been demonstrated by Zhang et al. [50] that continuous friction caused further spalling pits and grooves on the wear surface. The main wear mechanisms of low-carbon steel are abrasive and adhesive wear.

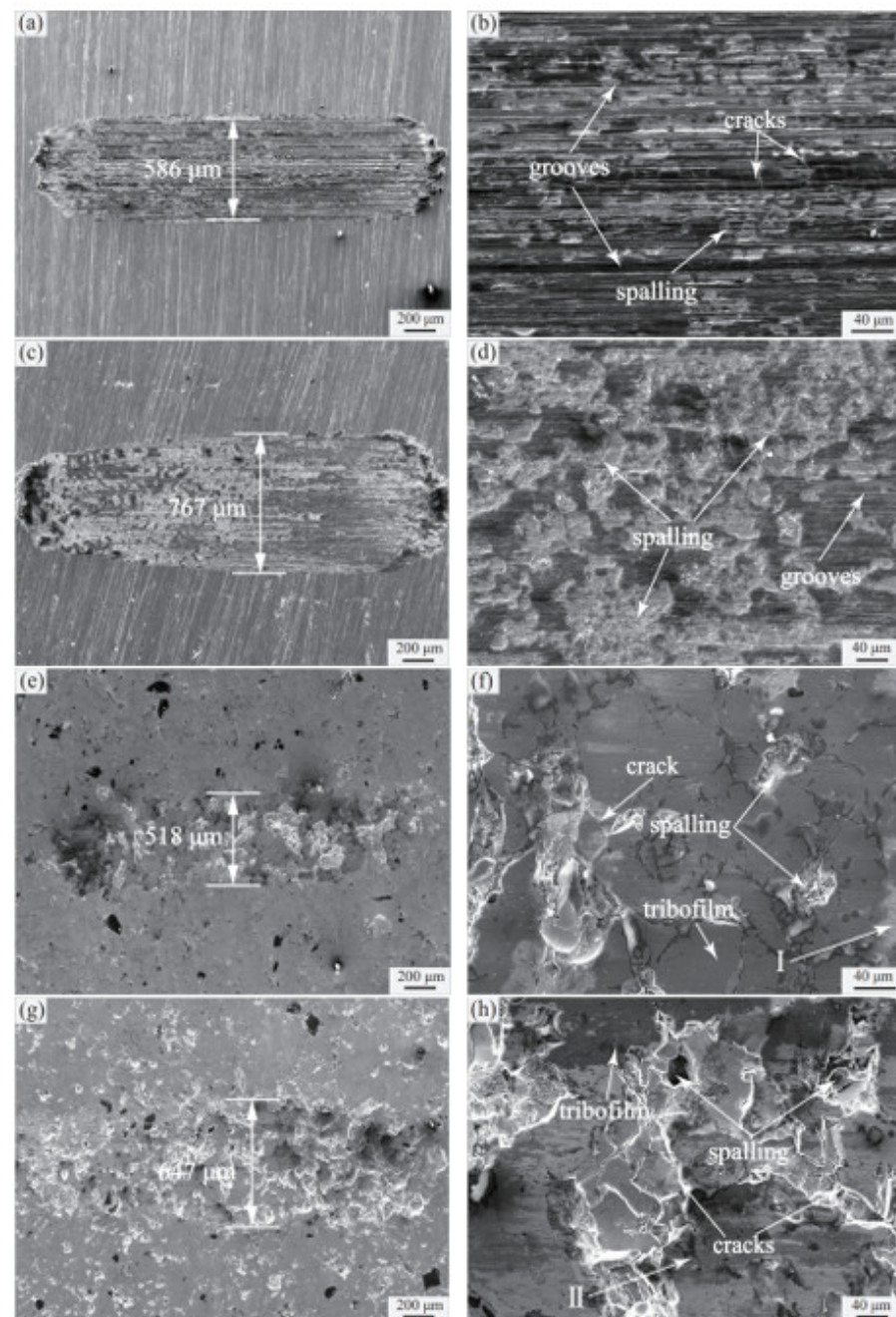


Figure 11. Micromorphology of wear scars of low-carbon steel and coating at sliding speeds of 500 t/min and 1000 t/min (a,b) low-carbon steel, 500 t/min; (c,d) low-carbon steel, 1000 t/min; (e,f) Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$, 500 t/min; (g,h) Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$, 1000 t/min.

Figure 11e–h show the micromorphology of the wear scars of the Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating at sliding speeds of 500 t/min and 1000 t/min. Obvious spalling and micro-cracks form on the worn surface of the coating. This is because the splat droplets interlocked each other during spraying, forming a mechanical bond. Under the continuous sliding action, the cracks propagated from the pores and defects between the coating layers. With the increase in the sliding speed, pores and cracks accumulated and expanded, and the coating shows a fatigue failure trend, eventually leading to deeper cracks and large-scale spalling on the surface. However, the ceramic particles embedded in the coating are more prone to spalling. These spalling ceramic particles are not discharged in time, and plastic deformation occurs at the friction contact point, eventually forming different spalling pits and pores in the coating. Table 9 shows that the elemental composition in grey area I and black area II contain more Fe and Cr elements, while black area II contains a small number of O elements. This is due to the oxidation reaction of the worn surface material caused by friction heat. A comparison of Figure 11f,h shows that when the sliding speed is 1000 t/min, the area of the oxide film in the black area II is smaller, which conforms to the above conclusion that the wear rate increases at a sliding speed of 1000 t/min. The main wear mechanism is the generation and propagation of cracks in the pores of the coating resulting in material spalling, accompanied by slight oxidative wear and abrasive wear.

Table 9. The results of EDS in different areas (wt. %).

No.	Main Element Content						
	Fe	Mn	Cr	Ni	Al	Si	O
I	74.94	7.74	14.57	2.18	0.01	0.56	-
II	66.74	5.57	13.20	1.30	0.31	3.46	9.42

4. Conclusions

In this study, Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ composite coating was fabricated on low-carbon steel by HVAS. The effects of different process parameters of spraying voltage, spraying current, and spraying distance on the porosity and microhardness of the coating were investigated by the orthogonal test method. The phase composition, microstructure, mechanical and tribological properties of the coating under the optimal spraying process parameters were studied:

- (1) The optimum spraying process parameters obtained were a spraying voltage of 41 V, a spraying current of 200 A, and a spraying distance of 150 mm.
- (2) The Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating comprised $\alpha\text{-Fe}$, (Fe, Cr) solid solution, Al_2O_3 , B_4C , and Fe_3B phases. The average porosity of the coating was $2.24 \pm 0.32\%$.
- (3) The average microhardness of the Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating was $1543 \pm 145 \text{ Hv}_{0.1}$, which was 8 times higher than that of the low-carbon steel. This was mainly due to the solid solution strengthening of the (Fe, Cr) solid solution phase and the diffusion strengthening of Al_2O_3 , B_4C , and Fe_3B hard phases.
- (4) Under the same load, the COF of Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating and low-carbon decreased with the increase of sliding speed, and the COF of the coating was lower than that of low-carbon steel. At the sliding speeds of 500 t/min, 750 t/min, and 1000 t/min, the wear rates of the coatings were $6.6 \times 10^{-6} \pm 0.8 \text{ mm}^3/\text{N}\cdot\text{m}$, $8.4 \times 10^{-6} \pm 1.5 \text{ mm}^3/\text{N}\cdot\text{m}$ and $12.7 \times 10^{-6} \pm 1.1 \text{ mm}^3/\text{N}\cdot\text{m}$, respectively, which were 65%, 70% and 63% lower than those of the low-carbon steel. The main wear mechanism of Fe-based- $\text{Al}_2\text{O}_3\text{-B}_4\text{C}$ coating was material spalling, accompanied by slight oxidative wear and abrasive wear.

Author Contributions: Conceptualization, H.L. and F.H.; methodology, H.L. and M.K.; software, F.H.; validation, J.L. (Jinran Lin), J.L. (Jitao Liu) and J.N.N.; formal analysis, H.L., J.N.N. and J.L. (Jitao Liu); investigation, F.H., M.K. and J.L. (Jinran Lin); data curation, H.L. and F.H.; writing—original draft preparation, H.L.; writing review and editing, J.N.N.; supervision, J.N.N. and J.L. (Jitao Liu); project administration, J.L. (Jinran Lin) and M.K.; funding acquisition, M.K. All authors have read and agreed to the published version of the manuscript.

Funding: This present work was supported by the Fundamental Research Funds for the Central Universities (No. XUEKEN2022016), Jiangsu provincial Key Research and Development Plan (No. BE2020311), and the National Natural Science Foundation of China (No. 52109091).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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