

Article

Effect of CeO₂ Content on Microstructure and Wear Resistance of Laser-Cladded Ni-Based Composite Coating

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Abstract: In order to improve the wear resistance of 45 steel, in this study, WC/Ni60 composite coatings with different CeO₂ additions (0, 1, 2, and 3 wt%) were prepared on 45 steel by the laser cladding technique; the experimental analysis was carried out by means of scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), X-ray diffraction (XRD), a Vickers hardness tester, and a friction and wear tester. The results show that CeO₂ had little effect on the phase composition of the coatings; however, with the increase in CeO₂ content, the CeO₂ played a key role in refining the grains of the coating, thus reducing the generation of cracks. In addition, CeO₂ could effectively strengthen the internal structure of the coating and improve its microhardness and wear resistance. Particularly noteworthy is the observed reduction in both the friction coefficient and mass loss of the coating when the CeO₂ addition reached 2%. This suggests an enhancement in the tribological performance of the coating at this concentration.

Keywords: laser cladding; rare-earth oxides; microstructure; grain refinement; wear resistance



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1. Introduction

To date, 45 steel has been widely used in machinery manufacturing, automobiles, construction, mining equipment, and other fields because of its commendable mechanical properties, processing performance, and low cost [1–4]. However, the wear resistance of 45 steel is insufficient, meaning it may not meet the criteria in some application scenarios requiring high wear resistance and results in the premature wear of parts. In addition, its fatigue performance is also limited. Therefore, the surface modification of 45 steel is of great significance.

With its excellent high hardness and strength advantages, ceramic materials have achieved a prominent position in the field of reinforcement and repair of key mechanical equipment components [5]. Through the application of laser cladding technology, the constraints faced by ceramic materials in the processing process have been successfully overcome, thus achieving the preparation of high-quality ceramic coatings on metal surfaces [6]. For example, Hu et al. [7] prepared nickel-based fusion-coated coatings containing different WC additions on stainless steel substrates and observed inhomogeneity in the distribution of WC particles. Further analysis shows that with the increase in WC mass fraction, the degree of grain refinement and microhardness of the coating show an increasing trend. Xia et al. [8] investigated the role of adding WC particles with different particle sizes in nickel-based composite coatings. The results show that the average microhardness of the composites increases by 59.70% (coarse WC) and 74.66% (fine WC), and the wear rate

decreases by 84.87% (coarse WC) and 89.17% (fine WC), respectively. The study also elucidated that WC particles have physical shielding and solid solution strengthening effects. The WC reinforced Fe-based amorphous composite coating prepared using rectangular spot laser cladding technology by Zhou et al. [9] can significantly improve the surface quality and improve the microhardness after laser remelting.

Laser cladding technology is of great significance in improving the wear resistance of metal parts. It not only prolongs the service life of key components but also optimizes resource utilization. Hence, in recent years, researchers have employed rare-earth elements in ceramic coatings through laser cladding technology to further improve their performance. These results show that the introduction of rare-earth elements can have a positive impact on coating performance in many aspects. Firstly, rare-earth elements can refine the microstructure of the coating, enhance the fluidity of the molten pool, and promote the discharge of gas from the molten pool, thereby effectively reducing the porosity of the coating and improving the overall quality [10]. Secondly, by adding rare earths or rare-earth oxides (such as Y_2O_3 , CeO_2 , and La_2O_3) to the cladding powder, the cracking tendency of the coating can be further reduced, the formation of defects such as pores can be inhibited, the grains can be refined, and the microstructure uniformity of the coating can be improved [11]. For example, Quazi et al. showed that the moderate addition of rare-earth oxides (e.g., Y_2O_3 , CeO_2 , and La_2O_3) improves the appearance and microstructure of coatings, reduces the crack susceptibility and porosity, and effectively prevents defects, such as cracks, pores, and inclusions, from occurring during laser melting and cladding [12], which is of great significance for studying the mechanism of the role of rare earths in laser melting and cladding composite coatings. Ye et al. [13] found that the addition of CeO_2 has a significant effect on the microstructure, hardness, and corrosion resistance of a laser cladding coating on the surface of titanium alloy, and proper addition can improve the performance. Zhang et al. [14] studied the effect of Y_2O_3 addition on the quality, microstructure, and microhardness of a multi-pass laser cladding Ti-6Al-4V coating by the coaxial powder feeding method. The results show that the addition of Y_2O_3 can completely eliminate the formation of pores in the coating and improve the quality and hardness of the coating. At the same time, the introduced Y_2O_3 has a great influence on the grain boundary and grain, resulting in grain boundary strengthening and grain refinement.

Building upon prior domestic and international research, the primary objective of this study was to fabricate WC/Ni60/ CeO_2 composite coatings using laser cladding technology to enhance the microhardness and wear resistance of 45 steel surfaces. This research holds significant implications for the investigation of surface reinforcement techniques for critical components. Furthermore, the study investigated the impact of varying weight percentages of CeO_2 on the geometric morphology, microstructure, wear resistance, and hardness of the coatings. To achieve this, distinct CeO_2 content additives were formulated, followed by the execution of laser cladding experiments and comprehensive evaluation of the resultant coatings.

2. Test Materials and Methods

2.1. Test Materials

In this experiment, 45 steel was chosen as the base material, with a size of 50 (length) $\times$ 50 (width) $\times$ 5 (height) (unit: mm). Ni60 and WC powders of 300~500 mesh and CeO_2 powders of 500 mesh were mechanically mixed by a FP400 planetary ball mill (180 r/min, 1 h). The mixed powder was dried in a vacuum oven (200 °C, 2 h). In the comparative experimental design, the percentage of rare-earth oxide CeO_2 was set to 0%, 1%, 2%, and 3%, respectively. The CeO_2 powder is shown in Figure 1a, the WC powder is shown in Figure 1b, the Ni60 powder is shown in Figure 1c, and the chemical composition of 45 steel is shown in Table 1. The exact volume fraction of the powder is shown in Table 2.

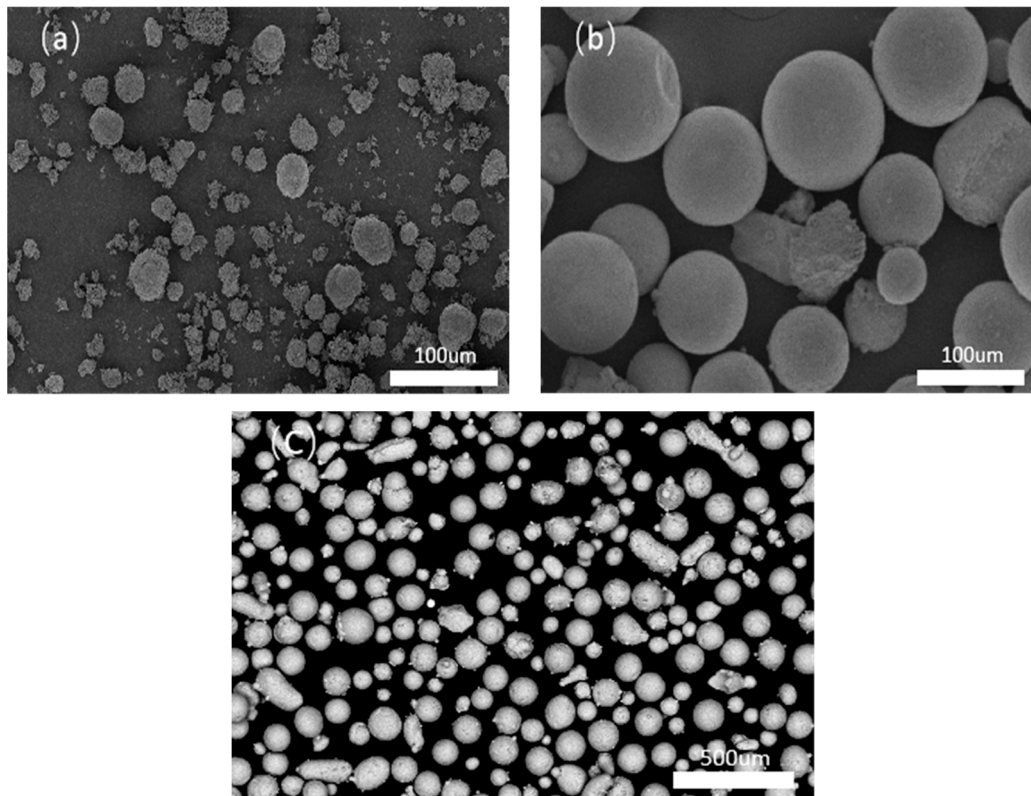


Figure 1. Morphology of cladding material: (a) CeO₂ powder; (b) WC powder; (c) Ni60 powder.

Table 1. Chemical composition of 45 steel (wt%).

Materials	C	Si	Mn	Ni	Cu	Fe
Value	0.42–0.05	0.17–0.37	0.5–0.8	≤0.30	≤0.25	Bal

Table 2. Ni60, WC, and CeO₂ component ratio (mass fraction, %).

Sample	A	B	C	D
Ni60	7	6.3	5.6	4.9
WC	3	2.7	2.4	2.1
CeO ₂	0	1	2	3

2.2. Test Method

The XL-F2000 W fiber laser cladding system was selected for the laser cladding experiments, and the maximum output laser power of the equipment is 2000 W. For the powder feeding method, we employed the prefabricated technique. Prior to experimentation, the substrate surface underwent sanding using sandpapers of varying specifications (240, 600, 800, 1500, 3000, and 5000 mesh) to eliminate surface oxides and impurities, followed by thorough cleaning with anhydrous ethanol to remove any residual debris and oil stains, and was subsequently air-dried in a ventilated environment. The mixed powder was uniformly applied onto the substrate surface at a thickness of 1 ± 0.1 mm. Through preliminary experiments, the optimum process parameters were determined as follows: a laser power of 1500 W, scanning speed of 10 mm/s, and defocusing amount of +5 mm, all of which induce a better cladding effect. Subsequently, a multi-channel laser cladding test was conducted using these established parameters. The schematic diagram depicting the laser cladding process is presented in Figure 2. Post-cladding samples underwent further processing including wire cutting (utilizing a Deep Yang QC350 K, Sichuan, China), inlaying, grinding, and polishing, after which the cross-sectional morphology of the coating was examined

using an XJL-302 optical metallographic microscope (Guangzhou, China). Additionally, the microstructure of the coating was analyzed using a JSM6460 scanning electron microscope (SEM) (Brno, Czech Republic). The elements and compounds of the composite coating were analyzed by an X-ray diffractometer (XRD-6100) produced by Shimadzu, Shimane-ken, Japan, and the radiation source was $\text{CuK}\alpha$. Furthermore, the microhardness of the cladding layer was tested by a digital microhardness tester (MHVD-1000AT) (Shanghai, China). The test was conducted with a 150 μm spacing from the top of the cladding layer to the substrate. Measurements are taken at three points, each spaced 50 μm apart on either side of the vertical test direction. The applied load was 200 g, and the duration of loading was 10 s. The wear resistance of the composite coating was tested at room temperature using the SFT-2M pin-on-disc friction and wear tester produced by Lanzhou Zhongke Kaihua Technology Co., Ltd. (Lanzhou, China). Dry sliding friction was tested. The grinding material was a Si_3N_4 steel ball with a diameter of 4 mm. The applied load was 40 N, the rotation speed was 200 r/min, the rotation radius was 2 mm, and the frequency was 1 Hz. The test time of each sample was 30 min.

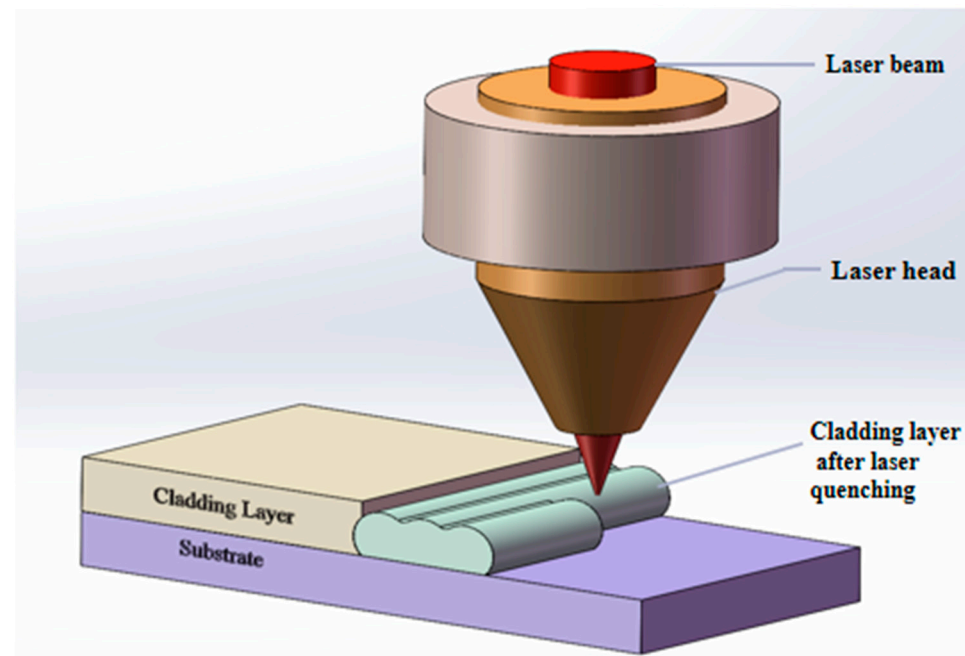


Figure 2. Schematic diagram of laser cladding processing.

3. Results and Discussion

3.1. Coating Macromorphology Analysis

Figure 3 presents the cross-sectional SEM images (a1–a4) and surface macrographs (b1–b4) of the laser-cladded Ni60/WC composite coatings with varying CeO_2 contents. From top to bottom are the obtained cladding coatings with CeO_2 mass fractions of 0%, 1%, 2%, and 3%. The results show that no defects such as cracks or pores were observed in the cross-sections of all samples, as shown in Figure 3(a1–a4). Figure 3(b1) shows that for the coating without CeO_2 , the surface is smooth, but there are long cracks. These cracks extend along the transverse direction and form an angle of nearly 90° with the cladding direction. Observing Figure 3(b2–b4), it is evident that the addition of CeO_2 resulted in a smoother surface for the coating, with no noticeable cracks. Given the susceptibility of nickel-based alloy coatings to crack, this outcome can be primarily attributed to the significant residual tensile stress concentrated within the coating and at the interface between the coating and the substrate [15], which commonly facilitates crack propagation. Therefore, the addition of cerium oxide (CeO_2) can optimize the forming quality of the coating.

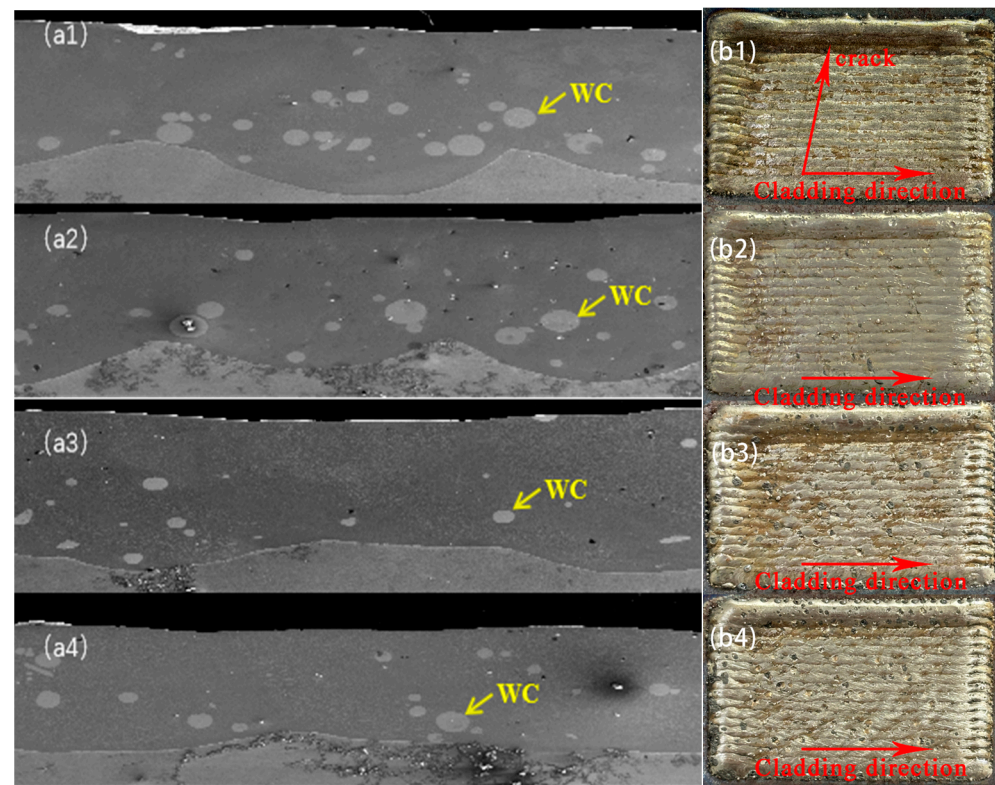


Figure 3. Macroscopic morphology of the coating: typical cross-sectional morphology (a1–a4) and surface macrograph (b1–b4).

3.2. Microscopic Morphology

Figure 4 shows the X-ray diffraction (XRD) patterns of Ni/WC composite coatings with different cerium oxide contents after laser cladding. The strong peaks of C, WC, and W₂C were observed for four different contents of cerium oxide, which verified the fact that WC can be synthesized in situ [16,17]. The existence of WC proves the existence of unmelted WC particles in the coating. The existence of W₂C phase indicates that during the solidification process, W reacts with C, resulting in non-equilibrium solidification during the cladding process [18]. Remarkably, with a cerium oxide content surpassing 2%, there is a notable increase in the quantity of W₂C hard phase. In addition, under intense laser irradiation, iron is considered to be diluted from the substrate due to high-temperature melting and liquid-phase flow, while the remaining tungsten is dissolved in the Ni matrix after the in situ reaction. Therefore, there is also a certain proportion of Fe_{0.64}Ni_{0.32} phase in the coating, which verifies the existence of the non-equilibrium solidification phenomenon in the cladding process again.

Based on the X-ray diffraction (XRD) analysis results of the aforementioned coatings, it is evident that all samples exhibit a similar crystal phase composition. Thus, to investigate the impact of CeO₂ on the coating, samples 0, 1, 2, and 3 were chosen for microstructural comparison. Moreover, the multi-channel coating was segmented into two regions (bottom region and middle region) to probe the microstructural evolution of the coating, allowing for a detailed analysis of the structural changes and hardness values within the coating.

Figure 5 shows the morphology of the bottom region of the coating with a different cerium oxide content. The grains at the bottom of the cladding layer show an obvious orientation, and the columnar crystals mainly extend along the direction perpendicular to the coating fusion line. This phenomenon is due to the slower heat dissipation rate at the bottom of the molten pool near the substrate, resulting in the growth of particles at the bottom of the coating along the top direction [19]. Before the addition of CeO₂, the grains at the bottom of the cladding layer are coarse, including large planar grains and columnar crystals. The addition of CeO₂ results in significant grain refinement [20,21]. With the increase in CeO₂ content, the grains at the bottom of the cladding layer are refined first and

then coarsened. When the CeO_2 content is 2%, the grain reaches the finest state, and when the CeO_2 content is 3 wt%, the grain becomes coarser.

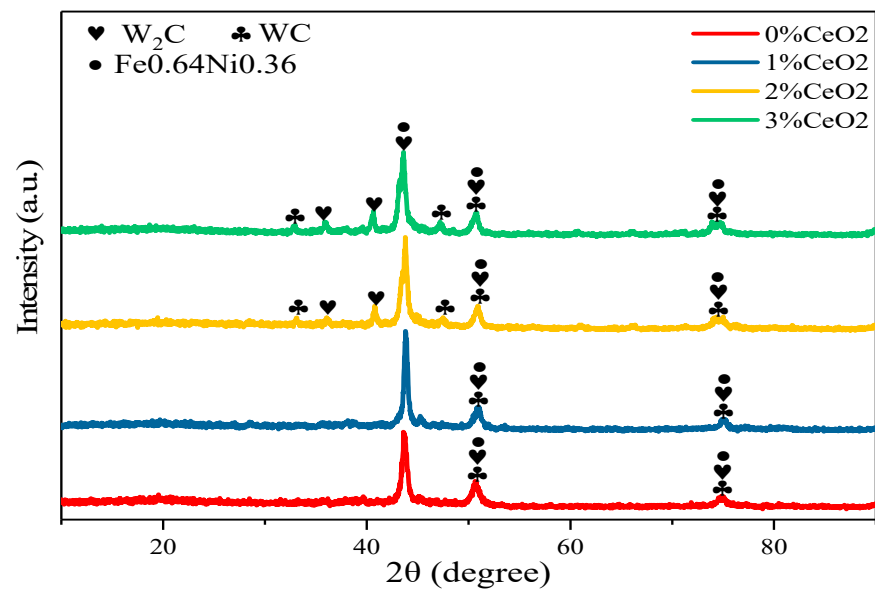


Figure 4. Phase composition of Ni/WC composite coating.

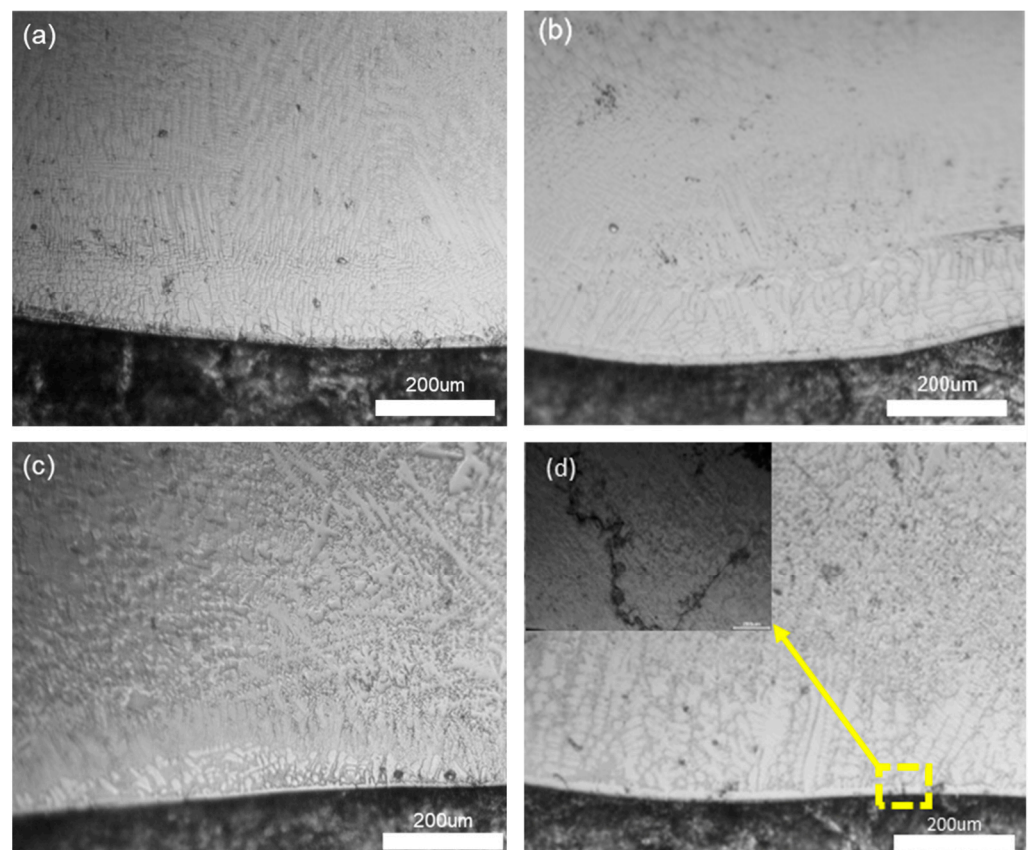


Figure 5. Microstructure of Ni/WC composite coating at the bottom: (a) S0; (b) S1; (c) S2; (d) S3.

Figure 6a–d shows the morphology of the middle region of the coating with different cerium oxide contents. Before the addition of CeO_2 , the grains in the middle of the cladding layer are mainly composed of larger dendritic and rod-like crystals. After the addition of CeO_2 , the WC particles in the middle of the cladding layer grow radially around [17],

which leads to the refinement of the grains around the WC particles, thus significantly enhancing the fine grain effect [22]. With the increase in CeO_2 content, the grains of WC particles in the middle of the cladding layer are refined first and then gradually coarsened. When the CeO_2 content is 2%, the most dendrites and secondary dendrites are observed around the WC particles.

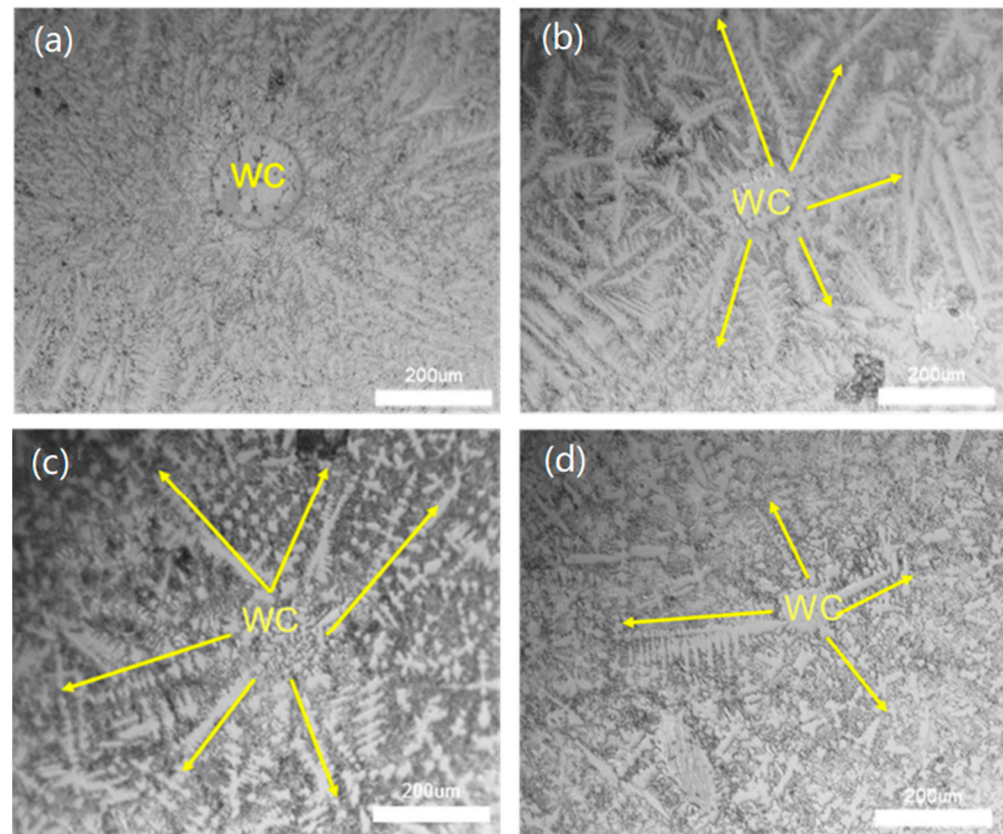


Figure 6. Microstructure of the middle part of Ni/WC composite coatings: (a) S0; (b) S1; (c) S2; (d) S3.

Cracks are evident in the magnified area of Figure 5d. The underlying reasons for this phenomenon are multifaceted: Firstly, the uniformity of the microstructure within the cladding layer emerges as a pivotal factor influencing cracking [20]. The excessive addition of CeO_2 to form refractory compounds diminishes the fluidity of the molten pool, exacerbating organizational stresses [21], thus precipitating crack formation. Secondly, during the solidification phase, the excess of rare-earth elements accelerates the absorption rate of the cladding material, thereby enhancing energy within the molten pool and facilitating the release of more WC particles [23]. The WC content is proportional to the hardness range; that is, the higher the WC content, the higher the hardness of the coating, but the worse the ductility. The precipitated carbides will increase the tensile stress, thus increasing the crack sensitivity [24,25]. Therefore, the higher WC content deposited at the bottom will also lead to cracks.

3.3. Hardness Analysis

Microhardness serves as a crucial parameter for assessing coating performance. Figure 7 illustrates the microhardness variation along the cross-section of the samples, spanning from the coating's apex to the substrate. As depicted in Figure 7, all sample coatings exhibited a notable increase in microhardness. Figure 8 illustrates a progressive increase in the average microhardness of the samples. Specifically, the microhardness of sample S2's coating reached 610.83 HV, approximately threefold that of the substrate (202.86 HV). Similarly, sample S3's coating achieved a microhardness of 634.63 HV, approximately 3.1 times greater than that of the substrate. The results demonstrate that the addition of CeO_2 to the coating led to a more pronounced increase in hardness compared

to the S0 sample. The results show that the addition of CeO_2 can improve the hardness of the coating. The reason for the higher hardness of the S1 and S2 samples is that the addition of a low content of CeO_2 makes the microstructure of the cladding layer denser and the grains gradually smaller [21]. The grain refinement and solid solution strengthening effect improve the strength and microhardness of the cladding layer [26]. Furthermore, the heightened surface hardness observed in sample S3 can be attributed to two primary factors: Firstly, Figure 3(a4) reveals a higher presence of unmelted WC particles in the middle and lower sections of the coating, contributing to enhanced coating hardness [16,17]. Secondly, the increase in WC content leads to the decomposition of WC into W and C atoms, subsequently dissolved within the matrix, thereby enhancing the solid solution strengthening effect [27,28].

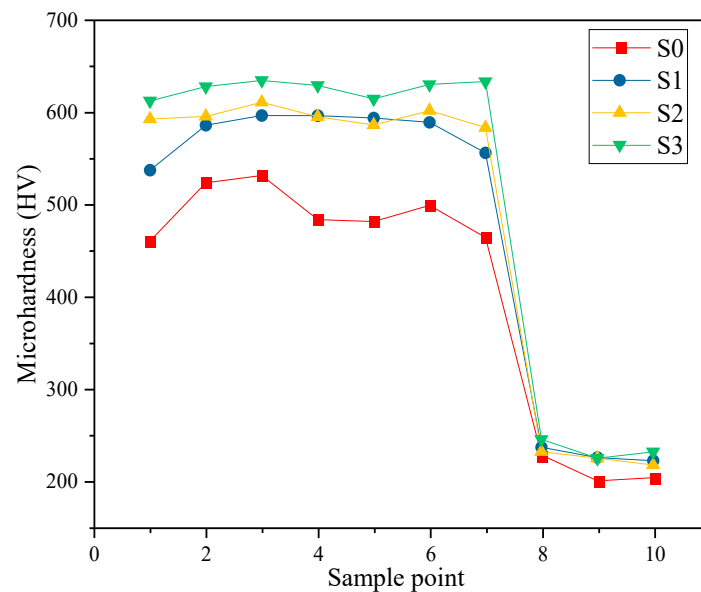


Figure 7. Microhardness distribution of Ni/WC composite coating.

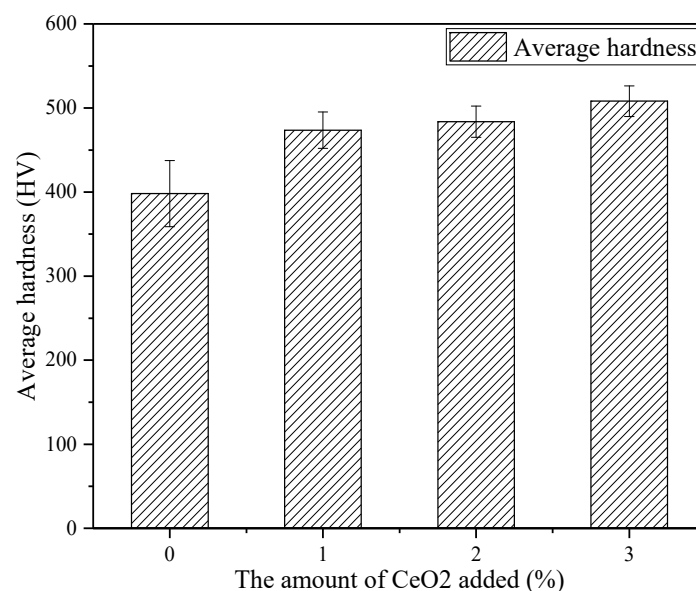


Figure 8. Average microhardness distribution of Ni/WC composite coating.

3.4. Wear Resistance Analysis

Figure 9 shows the variation curve of the friction coefficient of coating samples with different ratios. Under the load condition of 40 N, the sliding friction enters the stable wear stage after a run-in stage. Comparative analysis reveals a significant reduction in the average

friction coefficient of samples S0, S1, S2, and S3 relative to the substrate, indicative of superior wear resistance exhibited by the coatings. Notably, in comparison to sample S0 lacking CeO_2 , samples S1, S2, and S3 also demonstrate a noteworthy decrease in the average friction coefficient. Furthermore, the wear loss of samples S0, S1, S2, and S3 is also significantly reduced compared to that of the substrate, as depicted in Figure 10. The friction coefficient of the S0 sample exhibits considerable fluctuation within 20 min, indicating unstable wear resistance. This instability may stem from the presence of incompletely dissolved tungsten carbide particles within the coating. The interaction of these substances with the friction balls could induce violent friction phenomena [18]. Conversely, the friction coefficient curves of samples S1, S2, and S3 display a stable trend during the stable wear stage.

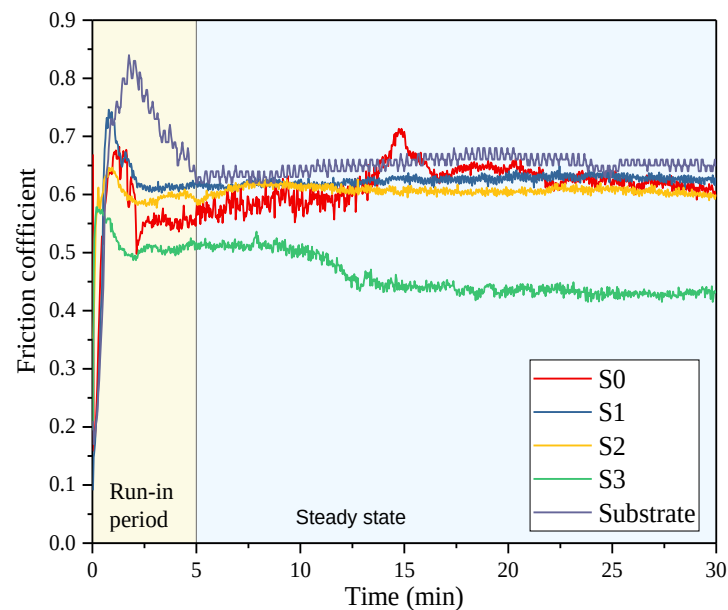


Figure 9. The friction coefficient curves of Ni/WC composite coatings with different samples.

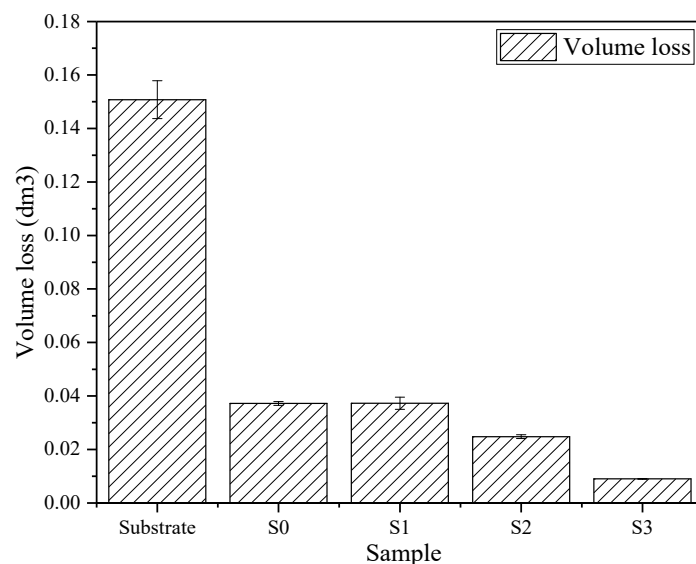


Figure 10. Wear loss of Ni/WC composite coatings with different samples.

In order to further study the wear mechanism of $\text{CeO}_2/\text{Ni60}$ coatings, the worn surface morphology of each sample in Figure 11 was observed by scanning electron microscopy (SEM). As illustrated in Figure 11a, the wear surface of sample S0 exhibits flake or substantial material shedding marks, along with the presence of plough-shaped grooves, indicative of abrasive wear and severe adhesive wear. Since the CeO_2 -free coating is softer than the

matched parts, the hard micro-convex bodies on the surface of the matched parts can easily penetrate into the sliding surface during the sliding process, forming a large wear contact area between the two. This leads to the formation of severe adhesive wear. Following the incorporation of CeO_2 , the wear surface of the coating manifests increased smoothness relative to the non- CeO_2 -incorporated counterpart, concomitant with significant reductions in both the friction coefficient and wear volume. Inspection reveals comparable wear degrees among samples S1, S2, and S3; nonetheless, furrows persist on the coating's wear surface, as depicted in Figure 11b–d. The heightened hardness of the coating impedes plastic deformation during interaction with mating components throughout the wear process, consequently diminishing wear contact areas. Moreover, the low plastic deformation contributes to the suppression of adhesive wear, aligning with Archard's well-known law of adhesive wear [29]. Therefore, the wear mechanism of the CeO_2 -added coating is mainly mild adhesive wear and abrasive wear [30]. This shows that the wear resistance of the coating with CeO_2 is significantly improved. The hardness of the coating material is proportional to the wear resistance of the coating [31]. The higher the hardness of the coating material, the better the wear resistance of the coating. The reason for sample S3 having the best wear resistance is as follows: First, the submicron CeO_2 particles are exposed to the surface of the friction pair during the wear process, providing lubrication [30]. Secondly, the WC particles on the coating surface are preferentially worn to protect the softer 45-steel substrate from direct contact with the Si_3N_4 steel ball. In addition, at high temperatures generated by dry sliding friction, broken WC decomposes into W and C elements. The new carbides formed by C, W, and other elements in the matrix are hard phases, which can significantly improve the hardness and wear resistance of the matrix [32]. The results show that the addition of CeO_2 can improve the wear resistance of the composite coating [27]. Combined with the above microstructure observation and hardness test, micro-cracks are formed inside the sample S3 coating. Therefore, when the added amount of CeO_2 is 2%, the tribological properties of the coating, such as the friction coefficient and wear rate, are superior.

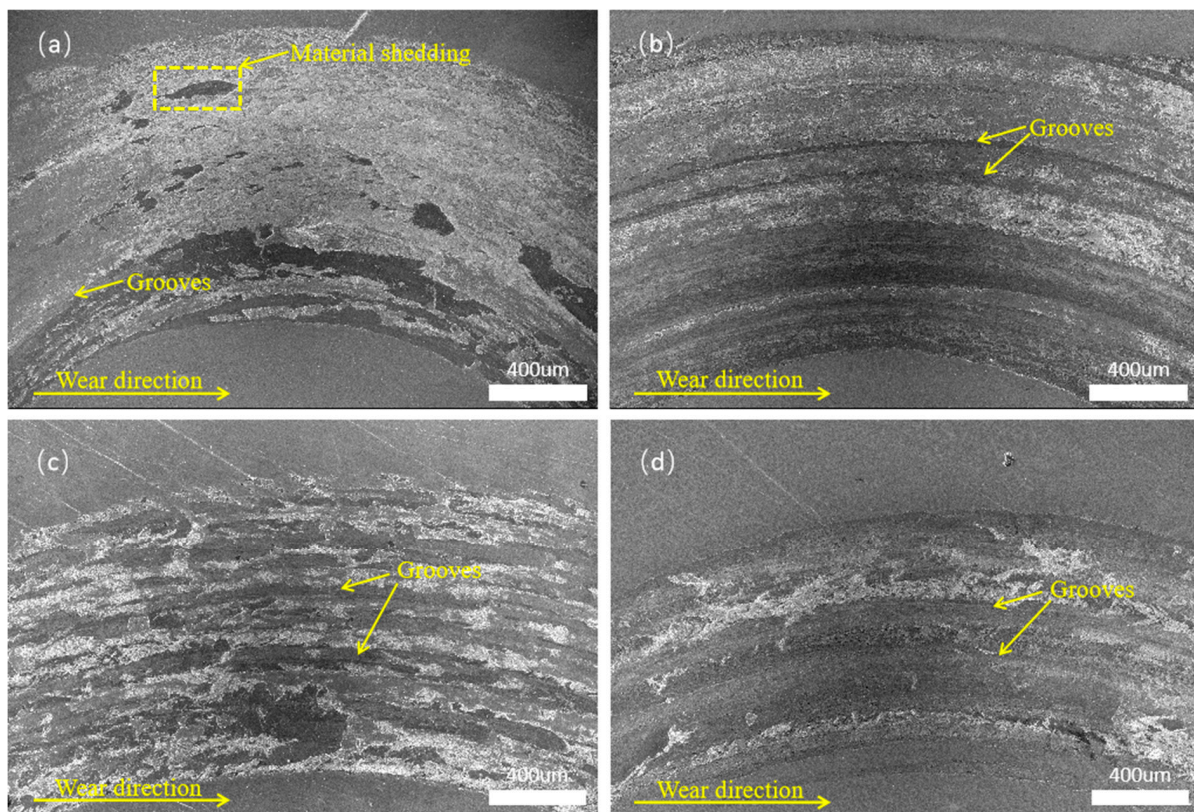


Figure 11. OM wear morphology of Ni/WC composite coatings for different specimens: (a) S0; (b) S1; (c) S2; (d) S3.

4. Conclusions

Ni60/WC/CeO₂ composite coatings were fabricated on the surface of a 45-steel substrate utilizing laser cladding technology. The surface modification effect of trace CeO₂ on the composite coatings was investigated and substantiated through an examination of macroscopic and microscopic features, hardness, and wear resistance. The principal findings are summarized as follows:

1. The incorporation of CeO₂ into the cladding powder demonstrated a capacity to mitigate surface cracks in nickel-based composite coatings, thereby enhancing the overall quality of the formed composite coating.
2. An optimal quantity of CeO₂ was shown to refine grain structures, promote uniformity in the microstructure of the cladding layer, and enhance solid solution strengthening within the cladding layer, thereby augmenting both the hardness and wear resistance properties of the coating.
3. When the CeO₂ addition reached 2%, notable enhancements in both the hardness and wear resistance of the composite coating were observed, resulting in optimized mechanical properties.

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

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