Effects of process parameters and carbon nanotubes content on microstructure and properties of laser cladding composite coatings using Ni-Ti-Cr-carbon nanotubes

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Abstract

The Ni-Ti-Cr-carbon nanotubes (CNTs) composite coatings was successfully fabricated by laser cladding. The optical microscope was used to observe the metallographic structure of the composite coating, the phase composition of the composite layer was detected by XRD, the microstructures of the composite coatings were observed by SEM, and the point distribution and line distribution of elements were analyzed by EDS. With the increase of laser specific energy (Es) and CNTs content, the TiC enhanced particles in the composite coatings evolves into coarse dendrites. Compared with the mild steel substrate, the microhardness and wear resistance of the composite coatings are obviously improved. The maximum microhardness obtained by the composite coating is approximately 5 times that of the mild steel substrate. The increase of Es and the excessive content of CNTs will reduce the microhardness and wear resistance of the composite coatings. The in situ synthesis of TiC particles not only enhance the microhardness of the composite coatings, but also improve the wear resistance of the coatings.

Mild steel is a well-known metal material with good comprehensive properties, and more importantly, low price, which is widely used in parts manufacturing, mechanized equipment and shipbuilding, etc [1, 2]. In the service period, parts (such as gears) and mechanical equipment (such as mining equipment) are very vulnerable to wear, which results in their performance degenerate or even scrap. Nevertheless, the relatively low hardness and wear resistance, which limits mild steel application area and service life. With the continuous development of laser cladding technology, laser cladding as a surface modification technology, by means of its unique technical advantages, has been widely applied in many fields, for instance aero-space, mechanical remanufacturing and repair, automobile industry, etc [3–5]. The coatings fabricated by laser cladding are provided with finer microstructure, controlled dilution rate, excellent metallurgical bonding with substrate [6]. Therefore, laser cladding is a competitive candidate technology to improve the wear resistance of mild steel, consequently, improving its usability.

The ceramic reinforced composite coating, manufactured by laser cladding, not only has favourable bonding ability with the substrate, but also grasps high hardness and superior wear resistance, which benefits from ceramic reinforced particles (CRPs) [7]. Thus it can be seen that the wear resistance of the composite coating is closely related to the CRPs. However, there is a great difference in the properties between different kinds of CRPs. Hence, the selection of CRPs possesses a great influence on the properties of composite coating. Titanium carbide (TiC) is an ideal reinforced particles in composite coating, due to its high hardness (3200 HV), high elastic modulus (2940 N/mm²), high melting point (3140 °C) and excellent chemical stability [8, 9]. Gopinath Muvvala et al [10] fabricated Inconel718/TiC metal matrix composite coating by laser cladding on AISI 304 austenitic steel. The composite coatings obtained outstanding wear resistance than the AISI 304 steel.
fabricated TiC/Fe-based composite coating with CeO$_2$ by laser cladding. They found that the wear resistance of composite coating had been improved obviously, and the shape, size, distribution, and existence form of the TiC particles had great influence on wear resistance of the composite coating. Hashem et al\cite{12} researched that the microstructure, wear resistance, and corrosion characteristics of laser cladding layers with TiC on low-carbon steel. The hardness of the layers was enhanced by about 19 times of the low-carbon steel, and the wear resistance of layers was reached to about 25 times of the substrate. After laser cladding, the corrosion resistance shows a incredible improvement. These previous studies suggest that TiC is an excellent CRPs in a composite coating.

There are two methods for adding TiC CRPs into the coatings, one is direct addition and the other is \textit{in situ} synthesis by the chemical reaction of titanium (Ti) and carbon (C). The \textit{in situ} synthesized TiC particles can acquire finer granularity, better binding ability with coating and more evenly distributed in the coating compared with the directly added TiC. Carbon nanotubes (CNTs) are one-dimensional carbon material with excellent electrical and chemical properties, which are widely used in the batteries and circuits\cite{13}.

Furthermore, the CNTs also possess excellent mechanical properties and are used in structural materials\cite{14–16}. Researchers have applied CNTs to the coatings by laser process. Yao et al\cite{17} researched the microstructure and hardness of laser cladding layers with carbon nanotube. The research results showed that the hardness of layers reached 900–1000 HV, which have obviously improved compared with the substrate (medium carbon steel). The wear resistance of carbon nanotube carburized layers was 2–3 times than the substrate. Wang et al\cite{18} studied manufacturing multi-wall carbon nanotube reinforced Inconel 625 composites by selective laser melting. They found that the carbonous composite acquired improvement in ultimate tensile and yield strength with less elongation. Chen et al\cite{19} reported laser powder deposition of carbon nanotube can enhance nickel–based superalloy Inconel 718. The experiment results showed that the tensile strength of the IN718/NiPCNTs coatings was obviously increased. Savalani et al\cite{20} reported that \textit{in situ} synthesized TiC by using Ti and carbon-nanotube by laser cladding on titanium alloy. The composite layers possess high hardness and excellent wear resistance because of the \textit{in situ} synthesized TiC reinforcements in layers. The above investigations revealed that CNTs and its \textit{in situ} synthesized ceramic as the enhancement phase can improve the hardness and wear resistance of laser processed coatings as the enhancement phase. In the experiments of titanium alloy as the substrate, the Ti elements that react with the CNTs to generate TiC come from the molten substrate, while in the experiments of mild steel as the substrate, Ti comes from the cladding materials. Distinctly, the two different sources of Ti lead to evident differences in the amounts of Ti elements that react with CNTs, which results in different contents and microstructure of \textit{in situ} synthesized TiC. Therefore, properties of the coatings are affected. However, it is regrettable that numbers of researches in laser cladding composite coatings using Ti and CNTs on mild steel are very rare.

This work fabricated TiC reinforced composite coatings by using Ni, Ti, Cr and CNTs powder with laser cladding. The objective of this experiment is to investigate the effect of CNTs contents on the microstructure and properties of the coatings. Moreover, the formation mechanism of \textit{in situ} synthesized TiC by Ti and CNTs, and the wear mechanism of composite coatings were mainly studied. This research will provide a technical reference...
for the applications of CNTs in the preparation of laser cladding coatings to improve the wear resistance of materials.

1. Materials and methods

In this experiment, Q235 mild steel hot rolled plates were utilized as the substrate with three-dimensional size 40 mm × 20 mm × 10 mm. Generally, prior to cladding the coating, the surface of mild steel plates should be polished off the rust by metallographic sandpaper. Before drying in vacuum oven at 110 °C for 2 h to remove the attached water, the polished plates were put into ultrasonic cleaning machine with deionized water for 15 min to wash wear debris. Ni, Ti, Cr and CNTs powder were selected as laser cladding powder. The morphologies and sizes of raw powders are exhibited in figure 1. To obtain the mixed powder, the pure powders of designed proportion was mixed evenly by a planetary ball mill with a rotation speed of 200 rpm for 3 h. In the process of powder mixing, the raw powders were mixed mechanically without chemical reaction. The mixed powders were dried in the vacuum drying oven at 110 °C for 2 h to remove the adsorbed free water. Before laser cladding process, the mixed powders were put on the substrate with 1 mm thickness. As the influence of process parameters on laser cladding composite coating was studied, the content of CNTs was fixed to 4 wt%. When the effect of CNTs content on the composite coating was studied, the laser specific energy was 66.7 J mm⁻².

TruDisk12003 disc type solid state lasers (λ = 1030 nm) was employed to manufacture the coatings. The laser output power was 600–1200 W, scanning speed was from 3 to 6 mm s⁻¹, and the laser beam diameter was set to 3 mm. The process parameters of laser cladding are shown in table 1. Argon gas was used as shielded atmosphere to protect oxidation of the molten pool, the flow rate of argon was maintained at 15 l min⁻¹.

After laser cladding process completed, the coatings were cut by wire-electrode from the transverse cross-sections. The phase compositions of coatings were detected via a Bruker D8 advance X-ray diffractometer (XRD) with Cu Kα radiation (λ = 0.1540598 nm) at 40 kV and 40 mA, and that step width is 0.02 degrees and scanning speed is 15 s tep⁻¹. After corrosion treatment with etching agent (HF:HNO₃:H₂O = 2:1:17 vol ratio) for 10 s, the cross-section morphology of coatings was observed through optical microscope (OM) and Field Emission scanning electron microscopy (FESEM) equipped with a Bruker Quantax400 energy dispersive spectrometer (EDS).
The micro-hardness of coatings, from nearby surface of the coatings to the substrate, were inspected in virtue of a HXS-1000A micro-hardness tester, with 200 g (1.96 N) load and 15 s dwelling time. Two adjacent test points are 0.1 mm apart. Wear test of substrate and cladding coatings were carried out by ML-10 friction and wear testing machine. 400# metallographic sandpapers were utilized as friction pair in this experiment.

Figure 3. The macroscopic morphologies of coatings surface with different content CNTs, (a) macroscopic morphologies of coatings surface with 2 wt% CNTs, (b) macroscopic morphologies of coatings surface with 6 wt% CNTs.

Figure 4. The morphologies of the coatings cross section with different Es.

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2. Results and discussion

2.1. Macroscopic morphologies of coatings

Figure 2 shows the macroscopic morphologies of 4 wt% CNTs coatings surface with different specific energy of laser (Es). Figure 3 shows the macroscopic morphologies of coatings surface with different content of CNTs. Having smooth surface, the coatings are well formed on the substrate surface without obvious crack defects. As seen in figure 2, with the increase of laser specific energy, the width of coating increases, which is because the higher specific energy makes more cladding materials enter the molten pool. From figure 3, the width of coatings increase with the increase of the amount of CNTs addition, which is due to CNTs increase the absorption of the laser, thus more cladding material is melted to form the coatings [21].

Figure 4 exhibits the morphologies of the coatings cross section with different Es. Un-bonding effect emerges at the edge of the coating with \( \text{Es} = 44.4 \text{ J mm}^{-2} \). The central energy density of the single-mode laser beam is high while the edge energy density of that is low. The laser energy obtained at the coating edge is lower when the Es is lower, so the coating edge cannot form an effective combination with the substrate. The fused cracks forms at the edge of coating at \( \text{Es} = 50.0 \text{ J mm}^{-2} \). When the molten pool solidifies and forms a coating, the volume will shrink, and the coating will be pulled by the substrate due to the difference between the thermal expansion coefficient of the coating and the substrate. When the Es is low, the temperature gradient between the coating and the substrate is large, triggering greater tension of coating subject from the substrate. Furthermore, cracks are generated after the tension is greater than the stress limit of the coating edge.

When Es is 66.7, 83.3 and 133.3 J mm\(^{-2}\), there are no un-bonding and cracked defects in the coatings. Additionally, an excellent metallurgical bond is formed between the coatings and the substrate. The metallographic structure of coating with 66.7 J mm\(^{-2}\) is demonstrated in figure 4. At the interface between the coating and the substrate, the laser energy is transferred to the substrate to reach the martensite transition starting temperature (Ms), thus martensite transformation occurred in the substrate to form the strip martensite. When the molten pool meets the substrate at room temperature, the liquid molten pool rapidly cools to form a large degree of subcooling and then form planar crystals eventually. After the formation of planar crystals, the temperature gradient (G) at the solidification front is high, but the solidification velocity (R) is low. The molten pool radiates heat along the direction perpendicular to the solid-liquid boundary, which can leads to the growth of columnar crystals in the opposite direction. As the solidification progresses, the G decreases and the R increases. There is no preferential heat dissipation direction in the molten pool, and the crystals grow into dendrites. On the upper part of the molten pool, especially on the surface of the molten pool, heat can be dissipated not only to the solidified coating, but also to the air. At this time, the temperature gradient is faster, and the grains grow into fine equiaxial crystals.

Figure 5 exhibits the macroscopic morphology of the cross section of coatings with various CNTs contents manufactured at Es of 66.7 J mm\(^{-2}\). There are no cracks and pores in the coatings with the addition of 2 wt% and 4 wt% CNTs, and the coatings are well bounded to the substrate. When the content of carbon nanotubes was 6 wt%, pores and cracks are observed in this coating. During laser cladding, the molten pool is protected from oxidation by protective gas, but CNTs have a large specific surface area and a good affinity between carbon and oxygen. When more CNTs (6 wt%) are added, there will be more opportunities for CNTs to contact with oxygen, so less CNTs are oxidized to form gas bubbles. Therefore, bubbles cannot escape from the pool before solidification, leading to the formation of pores in coatings. The energy distribution of the single-mode laser determines that the solidification speed of the edge of the molten pool is faster than that of the center of the molten pool. Therefore, bubbles on the edge of the molten pool escape from the molten pool in a shorter time than those in the center of the molten pool, so the coating edge is more prone to pores, as can be seen in...
Moreover, the diameter of pore near the coating center is obviously larger than that at the coating edge, because the bubble near the coating center have more time to float and grow up in molten pool.

2.2. Phase composition and microstructure of coatings

Figure 6 displays the phase composition of coatings with different Es. The coatings are mainly composed of TiC, Fe₂Ti, Cr₇C₃, FeNi, (Fe,Ni) and Cr. The process parameters have no obvious effect on the phase composition of the coatings. When the Es of the laser is low, CNTs cannot completely react with Ti in a very short time, so residual CNTs is found in the coating.

Figure 7 demonstrates the microstructure of coatings with different Es. From the figure 7, the coatings consist of grey matrix and black second phase. Moreover, the coatings microstructure shows uniform, dense and pores-free. As shown in figure 7(a1), the upper part of the coating is basically fine black point-like and strip-like second phase at Es = 44.4 J mm⁻². When Es = 50.0 J mm⁻², the morphology of upper part of coating is a small amount of black block and fine black strip-like second phase (figure 7(b1)). It is noted that the second phase of both coatings is very fine, even submicron second phases, because the molten pool cools rapidly at low Es, the second phase grains do not have sufficient time to grow after nucleation. Moreover, because the Es of the two coatings is close, the microstructure of the second phase is similar to that of the two coatings (figures 7(a1) and (b1)). When the coating with intermediate Es (66.7 J mm⁻²), the coating is dominated by black block phase, meanwhile, a small amount of point-like and flower-like phase in the coating (figure 7(c1)). With the higher Es
the microstructure of second phase in the upper part of the coatings (figures 7(a1) and (c1)) is obviously different from that of other coatings, which demonstrates that the dendritic or blocky second phase increases, the point second phase decreases distinctly, and the grain size of the second phase is significantly coarse. As seen in figures 7(a2), (b2), (c3), (d2) and (e2), the second phase in the middle of coatings is mainly black dendritic and blocky. Similar to the evolution law of the second phase microstructure in the upper part of the coating, the second phase in the middle part of the coatings also becomes coarse with the increase of the Es input. The second phase at the top of the coating is finer grain size than that at the middle. Moreover, when the Es is lower (44.4 and 50.0 J mm\(^{-2}\)), the microstructure of the second phase in the middle part of the coating is

| Phase | C   | Ti   | Ni   | Cr  | Fe  |
|-------|-----|------|------|-----|-----|
| 1     | 44.36 | 48.61 | 1.63 | 4.04 |
| 2     | 15.62 | 38.60 | 19.14 | 4.04 |
| 3     | 7.02  | 25.97 | 24.74 | 20.09 |
| 4     | 23.05 | 28.23 | 27.66 | 15.33 |
| 5     | 40.39 | 38.34 | 9.42  | 6.05  |
obviously different from that in the upper part. When the molten pool solidifies, the heat dissipation rate of the upper part of the molten pool is faster than that of the middle part of the molten pool. The second phase at the upper part of the coatings does not grow into developed dendrites after nucleation, while the second phase in the middle of the coatings has a longer time to grow into developed dendrites after nucleation.

In order to investigate the microstructure of coating with intermediate Es in further, the high magnification microstructure of the upper and middle parts of the coating with 66.7 J mm\(^{-2}\) is shown in the figures 7(c2) and (c4). From the figure 7(c2), obviously, this coating mainly consists of 3 different parts, namely, black blocky reinforcements (region 1), a lot of cellular eutectic phase (region 2) and a small amount of gray phase (region 3). Table 2 shows the chemical elements of the phase analyses by EDS. The black reinforcements (region 1 and 5) in coating mainly consists of Ti and C elements, and also contains a small amount of Ni, Cr and Fe elements, so it can be judged that the black phase is TiC. It should be emphasized that TiC in region 5 contains more eutectic
elements than TiC in region 1, because TiC density is small and easy to rise under the action of Marangoni flow. Therefore, so the TiC concentration at the bottom of coating is low and metal elements are more easily dissolved into TiC grains when TiC grains grow. The main constituent elements in the grain of coating matrix (region 2, 3 and 4) are Ti, Ni, Cr and Fe and less C, so it is clear that these regions are the metal matrix of the coating.

The black phase in figure 7(c5) is analyzed by element line scanning, in which the scanning path is along the white arrow. The element line scanning results of figure 7(c5) are shown in figure 8. When the scanning path passes through the black block phase, the curves of Ti and C elements rise, indicating that the black block phase is TiC. On both sides of TiC block, the curves of Ti, C and Fe gradually decrease or increase. Importantly, there is an obvious transition area, which indicates that TiC has good bonding property with the coating metal matrix.

Figure 9 shows the microstructure of the composite coating with 2 wt% (figure 9(a)) and 6 wt% (figure 9(b)) CNTs content manufactured at Es = 66.7 J mm\(^{-2}\). Figures 9(a1) and (a2) display low and high magnification microstructure of the upper part of composite coating with 2 wt% CNTs content respectively. Figures 9(b1) and (b2) display low and high magnification microstructure of the upper part of composite coating with 6 wt% CNTs content respectively. As can be seen from figure 9, with the increase of CNTs content, not only the content of in situ synthesized TiC increases, but also the TiC grain becomes coarse. The increase of TiC content in situ synthesis is beneficial to the growth of TiC grains, so the TiC grains tend to be coarse dendrites.

The cracks in the composite coating with Es = 50 J mm\(^{-2}\) were analyzed by SEM and element line scanning, and the results are shown in figure 10. In figure 10(a), the crack width at element line scanning position is about
10 μm, and that of the element line scanning position in figure 10(b) is about 6 μm. The cracks present a single extension without bifurcation crack. Combined with the metallographic picture of the cracks in figure 5, it can be seen that the cracks initiate from the top of the coatings and extend to the bottom of the coatings. It can be seen from figures 10(c) and (d) that the width of crack is significantly larger than the grain size of TiC and metal matrix. Moreover, there is no incredible difference in the distribution of elements on both sides of the crack, indicating that there is no difference between the phase composition on both sides of the crack. When the composite coating solidifies, tensile stress impacts tension stress, leading to the appearance of crack, when the tensile stress is greater than the tensile strength of the composite coating. Because the edge strength of composite coating is low, cracks mainly appear at the edge of composite coating.

The microhardness distribution of the composite coating prepared by different laser process parameters is shown in figure 11. Compared with the microhardness of the matrix, the microhardness of the composite coating is obviously improved, and the highest microhardness of the composite coating is about 5 times that of the metal matrix. With the increase of laser energy, the microhardness of composite coating decreases gradually. As the laser specific energy grows, Ti and CNTs have more time to in situ synthesize TiC, which is beneficial to improve the microhardness of the composite coating. At the same time, TiC grains tend to become coarse, which will weaken the strengthening effect of TiC on composite coatings. With the increase of the Es, the dilution rate
of the composite coating also increases. Consequently, more iron enters the composite coating, leading to the decrease of microhardness.

The microhardness distribution of the composite coating with different CNTs contents is shown in figure 12. When the content of carbon nanotubes is 2% and 4%, the microhardness of the composite coatings is similar. When the carbon nanotubes content is 6%, the microhardness of the composite coating decreases obviously. According to the XRD analysis results of the composite coatings, there are unreacted CNTs in the composite coating when the content of CNTs is 6%. Additionally, there are unreacted CNTs in the composite coating, and the TiC grains are coarse, which imposes a negative impact on improving the microhardness of the composite coating.

Figure 13 shows the worn surface morphology of substrate and composite coatings prepared by different process parameters. As can be seen from figure 13(a), the microhardness of the substrate is low, so the furrows on the substrate wear surface are dense and deep, which reflects the weak wear resistance of the substrate. Compared with the surface worn by the substrate, the furrows on the surface worn by the composite coatings are sparse and shallow, which indicates that the wear resistance of the composite coatings has been significantly improved. Figure 14 is the three-dimensional topography of the worn surface of the substrate and composite coatings. The wear degree of the wear surface can be seen more intuitively from the three-dimensional topography of the wear surface. The solidification of molten pool is very rapid during laser cladding process, which results in compact microstructure and fine grain of composite coatings. According to Hall-petch theory, the smaller grain size of the
material has the higher strength [22]. The fine grain size of composite coatings is beneficial to improve its wear resistance. TiC has high hardness, TiC reinforced particles can protect the composite coatings from abrasive wear. With the increase of light specific energy, the content of iron in the composite coatings increases, which not only reduces the microhardness of the composite coatings but also reduces the wear resistance of the composite coatings. Moreover, with the increase of laser specific energy, the grain size of TiC particles synthesized in situ becomes coarse, and the effect of precipitation strengthening is weakened.

Figure 15 shows the worn surface morphology of the composite coatings containing 2 wt% (figure 15(a)) and 6 wt% (figure 15(b)) CNTs. The worn surface morphology of the composite coating with 4 wt% CNTs content is shown in figure 13(d). Compared with the wear resistance of the substrate, the wear resistance of the composite coatings with different CNTs contents is obviously improved. As can be seen from figure 15, when the content of carbon nanotubes increases to 6 wt%, the furrows on the worn surface of the composite coating become dense and deep. This is because the excessive CNTs in the composite coating reduces its wear resistance.

3. Conclusions

In this work, the composite coating was prepared by laser cladding, and the microhardness and wear resistance of the coating were improved by in situ synthesized TiC from CNTs and Ti as reinforcement phase. In this research, the conclusions are as follows.
The Ni-Ti-Cr-CNTs composite coatings without defects are manufactured by laser cladding. The coatings are mainly composed of TiC, Fe2Ti, Cr2C3, FeNi, (FeNi) and Cr. Laser cladding parameters and CNTs content have little effect on the main phase composition of the composite coatings. Compared with the hardness of Q235 mild steel, the hardness of composite coatings enhanced dramatically. The maximum microhardness of the composite coatings is about 5 times that of the substrate. With the increase of Es, the microhardness of composite coating decreases. When the CNTs content is 6 wt%, the microhardness of the composite coating decreases obviously. The excessive Es and excessive CNTs (6 wt%) will deteriorate the wear resistance of the composite coatings.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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