



Article Optimization of the Forming Quality of a Laser-Cladded AlCrFeNiW_{0.2} High-Entropy Alloy Coating

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Abstract: Laser cladding is an effective surface strengthening method widely used in the surface treatment of extreme operating components such as gas turbines, aviation engines, and nuclear facilities. However, traditional cladding layers struggle to meet the diverse application needs of extreme working conditions due to their single cladding material and poor forming quality. Therefore, this article selected the new-type high-entropy alloy as the coating material and optimized its laser cladding process parameters in order to obtain an AlCrFeNiW_{0.2} high-entropy alloy coating with an excellent forming quality. It was found that as the laser power increased from 300 to 1800 W, the AlCrFeNiW_{0.2} high-entropy alloy coating transitioned from the incomplete or near-melted state to the fully and over-melted state gradually, while the coating showed the opposite trend of change as the laser scanning speed increased from 0.002 to 0.008 m/s. And when the laser power was 1000 W, the scanning speed was 0.005 m/s, and the spot diameter was 0.003 m, the AlCrFeNiW_{0.2} high-entropy alloy coating with a low dilution rate (9.95%) had no defects such as pores and cracks, and achieved good metallurgical bonding with Q235 steel substrate, demonstrating excellent forming quality. These could provide valuable theoretical and technical guidance for optimizing the laser cladding process and forming quality of new-type high-entropy alloy coatings.

Keywords: high-entropy alloy; coatings; laser cladding; process optimization; forming quality

1. Introduction

With the development of high-power laser technologies, laser cladding has been widely used in surface treatment of extreme working conditions such as gas turbines, aviation engines, nuclear facilities, and other components [1–4]. However, traditional cladding layers are difficult to meet the diverse application needs of extreme working conditions due to their single cladding material and poor forming quality. Thus, developing new laser cladding materials and efficiently optimizing their forming quality have become an urgent task at present.

The new-type high-entropy alloy material is the ideal laser cladding material, which has the excellent weldability, machinability, and strength toughness due to the unique four effects of the high-entropy effect, lattice distortion effect, slow diffusion effect, and cocktail effect [5–12], revealing outstanding advantages in surface modification, which has attracted many researchers' attention [13–19]. Qiu et al. [13] successfully prepared Al₂CrFeCo_xCuNiTi high-entropy alloy coating with good molding quality on the surface of Q235 steel by using laser cladding technology, taking laser power of 2500 W and scanning speed of 3 mm/s as the process parameter. Guo et al. [14] successfully prepared an FeCoNiAlTiCrSi high-entropy alloy coating on the surface of Q235 steel using laser cladding technology. The results showed that under the optimized laser process parameters of 1380 W power, scanning speed of 6 mm/s, overlap rate of 36%, and protective gas velocity



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of 1.5 L/min, the high quality FeCoNiAlTiCrSi coating with a thickness of 2 mm and no cracks and pores was prepared. Wang et al. [15] successfully prepared a (CoCrFeNi)₉₅Nb₅ high-entropy alloy coating on a Q235 steel substrate using laser cladding technology; the appropriate laser cladding process parameters were obtained and the controllability of the laser cladding process parameters for the preparation of high-quality high-entropy alloy coatings was proven. Researchers have conducted various studies on the optimization of the laser cladding process parameters and achieved many important research results. However, there is still a lack of systematic research on the relationship between the optimization of the laser cladding process and the forming quality of high-entropy alloy coatings, which makes it necessary to explore this in more detail.

Therefore, this article used the new-type AlCrFeNiW_{0.2} high-entropy alloy as the coating material and optimized its laser cladding process parameters. Orthogonal experiments of two factors and nine levels, with laser power (300, 800, and 1000 W) and scanning speed (0.002, 0.005, and 0.008 m/s) as factors, were conducted. Based on the analysis of morphologies and dilution ratio, the effects of the laser power and scanning speed on the forming quality of AlCrFeNiW_{0.2} high-entropy alloy coatings were deeply explored; thus, an AlCrFeNiW_{0.2} high-entropy alloy coating with an excellent forming quality was further obtained. This study could provide valuable theoretical and technical guidance for the optimization of the laser cladding process and forming quality of new-type high-entropy alloy coatings.

2. Materials and Methods

2.1. Experimental Materials

Q235 steel with a size of 15 mm \times 10 mm \times 80 mm was selected as the substrate material in this article; its composition is shown in Table 1. Before laser cladding, the substrate surface was polished with #240 sandpaper to remove the oxide layer, and then it was ultrasonically cleaned in alcohol and dried using a hair dryer. The raw materials were industrial pure Al, Cr, Fe, Ni, and W metal powders sold by Beijing Guanjinli New Materials Technology Co., Ltd. (Beijing, China). The purity was over 99.97% and the particle diameter was about 75 \pm 20 µm. According to the proportion of elements in the high-entropy alloy coatings, the metal powder was first weighed with a high-precision analytical balance (precision 0.0001 g) and then it was dry ground for 1h with an SFM-1 planetary ball mill at 175 rpm until it was evenly mixed. In order to remove and reduce impurity pollution, Al₂O₃ ceramic material grinding tanks and grinding balls were selected in this work. The mass ratio of the ball to material was 5:1 and the grinding ball diameters were 3, 5, 9, 12, and 15 mm, respectively. Afterwards, the obtained powder was placed in a vacuum drying oven at 100 °C for 3 h to remove excess moisture for later use.

Table 1. Compositions of Q235 steel (wt. %).

Element	С	Si	Mn	S	Р	Cr	Ni	Fe
Content	14.37	11.00	40.70	1.37	1.80	•••		Balance

2.2. Experimental Equipment and Method

The pre-laid laser cladding method was used to prepare the high-entropy alloy coating samples. Firstly, the pre-mixed and uniform powder was placed on the surface of the substrate using a fixture to form a powder bed with a thickness of approximately 1.2 mm. Then, the laser cladding was carried out under high-purity argon gas (flow rate $5 \text{ L} \cdot \text{min}^{-1}$) (LDF-4000-100, laser line, Laserline, Oelde, Germany); the principle schematic diagram is shown in Figure 1.



Figure 1. Schematic diagram of pre-laid laser cladding.

In addition, it is well known that the molten pool formed during the initial stage of laser cladding is formed under the combined thermal action of powder material, matrix, and heat source within the range of the laser spot size. This means that an appropriate spot size is beneficial for improving the efficiency of the coating preparation. At present, most of the reported laser cladding experiments choose 0.003 m spot diameter [3,20,21], so this size of laser beam was used in the laser cladding experiment of the AlCrFeNiW_{0.2} high-entropy alloy coating in this work. At the same time, laser power (300, 800, and 1000 W) and scanning speed (0.002, 0.005, and 0.008 m/s) were selected as factors for conducting orthogonal exploration experiments with two factors and nine levels, as shown in Table 2.

Table 2. The parameters of the orthogonal experiment.

Samples	А	В	С	D	Е	F	G	Η	Ι
P (W)	300	1000	1800	300	1000	1800	300	1000	1800
v (m/s)	0.002	0.002	0.002	0.005	0.005	0.005	0.008	0.008	0.008

The surface morphologies of the high-entropy alloy coating were realized using a field emission scanning electron microscope (SEM, Zeiss Supra 55, Carl Zeiss, Jena, Germany), and its built-in energy spectrometer (EDS, Carl Zeiss, Jena, Germany) was used to qualitatively analyze the composition content of the samples. Before testing, the surface of the high-entropy alloy coating samples should be polished with #240, #400, #800, #1000, #1200, and #2000 sandpapers in turn, and polished using a synthetic diamond polishing paste with a W0.5 grain size.

3. Results and Discussion

3.1. Topography Analysis

Figures 2 and 3 show the macroscopic and cross section morphologies of the AlCrFeNiW_{0.2} high-entropy alloy coating in the orthogonal experiment, respectively (sample G was not fully formed and the cross-sectional appearance could not be obtained, so it is not shown in Figure 3). It can be seen in Figures 2 and 3 that the laser power and scanning speed had a very close relationship with the coating forming effect. When the laser power was low and the scanning speed was too fast (such as G and H: P = 300/1000 W; v = 0.008 m/s), the lower laser energy was not enough to fully fuse the metal powder and substrate material; this resulted in an intermittent cladding layer and rough surface, and metallurgical bonding could not be achieved. In particular, cracks appeared in sample H (Figure 3), because when the laser power was low and the laser scanning speed was too fast, the cooling rate near the

melting point was too high, and the heat loss rate of the cladding layer was fast, resulting in insufficient heat and energy. This led to insufficient melting of the metal powder, and its deoxygenated slag components were not easy to float during the cladding process; thus, slag inclusions were formed, which seriously affected the metallurgical bonding between the cladding layer and substrate, resulting in the formation of cracks [22,23]. So, these parameters were not applicable to the formation of the AlCrFeNi $W_{0,2}$ high-entropy alloy coating. With the increase in laser power and the decrease in scanning speed, especially for sample E (P = 1000 W; v = 0.005 m/s), the alloy powder was melted and spread fully; the surface of the cladding layer was smooth and shiny, and showed a uniform width and height, indicating that the laser output energy was reasonable, which was suitable for the cladding work of the AlCrFeNi $W_{0,2}$ coating. However, when the laser power reached 1800 W and the scanning speed was reduced to 0.002 and 0.005 m/s (samples C and F), the excessive laser energy accelerated the liquid phase flow of the molten pool, resulting in a large number of agglomerated spheroidalized particles emerging at the edge of the coating layer, and the surface color became abnormal blue and purple, which was caused by excessive burning or even the volatilization of the metal elements [24]. It is for this reason that the coating exhibited a bumpy surface. Meanwhile, the cross-section diagram of sample C (Figure 3) showed a hole defect of about $30-40 \ \mu m$. More specifically, the reasons for these phenomena are as follows. In the case of high laser energy, too much of the substrate material was melted, and a large amount of dissolved gas was released in the molten pool, resulting in the formation of holes during the melting process. Furthermore, the melting of the metal powder was also accompanied by the generation of bubbles, which floated more on the surface of the molten pool and formed a rough and uneven coating surface after blasting under the action of fast cooling.



Figure 2. Macro morphology of the AlCrFeNi $W_{0.2}$ HEA coating corresponding to each process parameter under the orthogonal test.

Based on the above analysis, the cladding situation of the coating was preliminarily classified into four categories: insufficient melting (G, H), near melting (A, D, I), complete melting (E), and over melting (B, C, and F). As the laser power increased, the coating transitioned from the insufficient or near-melting states to the complete or over-melting states. While the coating exhibited the opposite trend of transformation with the increase in laser scanning speed. In addition, it should be pointed out that the surfaces of samples B, E, and I exhibited significant "ripple-like" characteristics, as shown in Figure 2, which were mainly caused by convection phenomena in the molten pool driven by surface tension gradients. That is to say, during the movement of the laser beam, the corresponding

front-end powder was rapidly melted, while the back-end powder produced a convective effect, as shown in Figure 4. This caused the protrusion of the liquid surface at the back of the molten pool, and it was quickly "frozen" as the rapid cooling process progressed, presenting the "ripple-like" morphology that bent in the opposite direction along the laser movement.



Figure 3. Sectional views of AlCrFeNi $W_{0.2}$ HEA coatings corresponding to different process parameters under orthogonal test.



Figure 4. "Ripple-like" morphology formation: (a) typical morphology and (b) schematic diagram.

3.2. Dilution Rate Analysis

In addition to the analysis of the sample morphologies mentioned above, dilution rate is another important manifestation of the coating forming quality, which is necessary in order to conduct detailed research. During laser cladding, the substrate material elements inevitably diffuse towards the coating direction, which inevitably leads to changes in the coating composition, known as the "dilution phenomenon". Researchers usually determine the degree of change in the composition of the cladding layer by calculating the size of the "dilution ratio". The calculation formula is as follows [25]:

$$\eta = \frac{A_{\rm c}}{A_{\rm s} + A_{\rm c}} = \frac{1}{A_{\rm s}/A_{\rm c} + 1} \tag{1}$$

Among them, A_s is the cross-sectional area of the coating and A_c represents the crosssectional area of the melting zone in the substrate. The changes in these two parameters can be more intuitively reflected in the coating (D) and substrate melting depth (H) [25], so Equation (2) is represented by parameters D and H, as follows:

1

$$\eta = \frac{D}{H+D} = \frac{1}{\frac{H}{D}+1}$$
(2)

The "dilution phenomenon" is a double-edged sword. On the one hand, it is a prerequisite for ensuring good metallurgical bonding between the coating and substrate, playing the role of component gradient transition, ensuring the stress generated during the melting process is released, and, to a certain extent, inhibiting the initiation of cracks. On the other hand, the dilution ratio has a direct impact on the structure and performance of the coating. If the value is too small, it can easily lead to poor adhesion between the coating and the substrate, while if it is too large, it can cause excessive deviation in the coating composition, which is not conducive to the display of its own characteristics. A low dilution rate could easily cause poor adhesion between the coating and the substrate, while an excessively high dilution rate would cause excessive deviation in the coating composition, which is not conducive to the manifestation of the coating's own characteristics. Therefore, under the premise of good metallurgical bonding, the dilution rate largely retained.

The dilution ratios of samples A–I were calculated using Equation (2) based on Figure 3 (where sample G was not fully formed and the cross-sectional appearance could not be obtained, so it was ruled out). The results are shown in Figure 5. In the case of low laser power and high scanning speed, low laser energy was not enough to cause deep melting on the substrate material, and the corresponding incomplete melting sample H showed the lowest dilution rate of 1.06%. The cross-sectional view (Figure 3) showed that sample H had a poor adhesion to the substrate, and some obvious cracks even appeared in the bonding area. So, the corresponding laser cladding process parameters of sample H were not suitable, although its EDS results were more similar to the nominal composition. As the laser power increased and the scanning speed decreased, the laser energy gradually increased, and the dilution rates of the samples also increased; meanwhile, the coating and substrate material gradually tended to have good metallurgical bonding. Correspondingly, the coating samples underwent a transition from near melting (A, D, and I) to complete melting (E). Among them, the dilution rate of the latter was 9.95% and the substrate and coating achieved good metallurgical bonding. In Table 3, it can be seen that the composition of the coating area was basically similar to the nominal composition, which once again confirmed that the process parameters corresponding to E were the most reasonable. As the laser power and scanning speed further increased and decreased, respectively, the excessive laser energy generated an excessive melting depth, resulting in the phenomenon of excessive dilution rates in the corresponding over-melted samples (B, C, F). Especially, the dilution rate of sample C was as high as 55.48%, and the Fe element content in the coating area was 51.29 at.%, which was more than twice the nominal content (23.81 at.%). In addition, Al elements with lower melting points were prone to problems such as burning loss and volatilization under high laser energy, resulting in the Al element content (15.79 at.%) being much lower than its nominal content (23.81 at.%). These all led to a significant deviation between the coating area composition and the nominal composition of the over-melted sample, which was not conducive to the display of the coating's own characteristics.



Figure 5. Dilution rates of the samples corresponding to different process parameters under orthogonal test.

Table 3. EDS results of the coating areas of the orthogonal experiment AlCrFeNiW_{0.2} high-entropy alloy samples (at.%).

Samples	Al	Cr	Fe	Ni	W
Nominal	23.81	23.81	23.81	23.81	4.76
А	23.78	23.83	23.93	23.74	4.72
В	21.12	18.03	40.13	18.13	2.59
С	15.79	15.16	51.29	15.19	2.57
D	23.19	23.86	24.48	23.78	4.69
E	22.88	23.79	24.77	23.78	4.78
F	16.09	22.73	36.95	22.01	2.22
Н	23.79	23.80	23.83	23.83	4.75
Ι	22.12	25.47	24.62	24.50	3.29

Overall, high-quality laser cladding coatings typically have the following characteristics, such as an intact overall morphology, high adhesion between the coating and the substrate, and reasonable coating composition. Based on the above analysis results, the optimal process parameters of the AlCrFeNiW_{0.2} high-entropy alloy coating were a laser spot diameter of 0.003 m, laser power of 1000 W, and scanning speed of 0.005 m/s.

4. Conclusions

In order to obtain AlCrFeNiW_{0.2} high-entropy alloy coatings with an excellent forming quality, this paper conducted orthogonal experiments with two factors and nine levels, taking laser power (300, 800, and 1000 W) and scanning speed (0.002, 0.005, and 0.008 m/s) as factors. Based on the analysis of morphologies and dilution ratio, the effects of laser power and scanning speed on the forming quality of the AlCrFeNiW_{0.2} high-entropy alloy coating were deeply explored. The following results were obtained:

- 1. For laser power P = 300/1000 W and scanning speed v = 0.008 m/s, the AlCrFeNiW_{0.2} high-entropy alloy coatings were in an insufficient melting state. For laser power P = 300 W and v = 0.002/0.005 m/s, and P = 1800 W and v = 0.008 m/s, the coatings were in a near-melting state. For laser power P = 1000 W and v = 0.002 m/s, as well as P = 1800 W and v = 0.002/0.005 m/s, the coatings were in a state of over melting.
- 2. As the laser power increased from 300 to 1800 W, the AlCrFeNiW_{0.2} high-entropy alloy coating transitioned from an insufficient state or near-melting state to the complete and over-melting states gradually, while the coating showed the opposite trend of change as the laser scanning speed increased from 0.002 to 0.008 m/s.
- 3. When the laser power was 1000 W, the scanning speed was 0.005 m/s and the spot diameter was 0.003 m, the AlCrFeNiW_{0.2} high-entropy alloy coating was in a complete melting state. In addition, the coating with a low dilution rate (9.95%) had no defects such as pores and cracks, and achieved good metallurgical bonding the with Q235 steel substrate, demonstrating an excellent forming quality.

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