

Research on the Forming Quality of CoCrFeNi-based High Entropy Alloy Coatings Prepared by Laser Cladding

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Abstract

This study is based on the CoCrFeNi alloy system. By adding alloying elements such as Al, Nb and Ti, and using laser cladding technology for preparation, it aims to optimize the composition design of alloy powders and the parameters of the cladding process. By optimizing the key process parameters such as laser power, scanning speed and powder feeding rate, the forming quality of the clad-ding layer was significantly improved, the generation of pores, cracks and other defects was reduced, and the influence of adding Al, Nb and Ti elements on the forming quality of the alloy was further explored.

Keywords

CoCrFeNi high-entropy alloy; Laser cladding; Forming quality

1. Introduction

In the nuclear power equipment system, key components (such as in-reactor components, pump and valve parts, etc.) are the core elements to ensure the long-term safe operation of the system, and their surface service performance directly affects the reliability and service life of the nuclear power plant [1]. At present, as nuclear power technology evolves towards extreme working conditions such as high temperatures and strong corrosion, traditional alloy coatings are no longer able to meet its increasingly strict service requirements. Laser deposition of high-entropy alloy coating technology provides an effective path to enhance the wear resistance and corrosion resistance of component surfaces, and has become a key means to extend the service life of critical components and promote the localization of nuclear power equipment [2].

In recent years, high-entropy alloys (HEA) have demonstrated remarkable application potential in industrial manufacturing, resource development, aerospace and

other fields due to their unique multi-principal near-isomeric miscibility properties [3]. The cocktail effect produced by the combination of multiple elements makes high-entropy alloys perform better than conventional alloys in terms of mechanical properties [4], corrosion resistance [5], and high-temperature oxidation resistance [6]. Among numerous high-entropy alloy systems, the CoCrFeNi series HEA exhibits excellent performance controllability due to the high matching degree of atomic radii and electronegativity of its constituent elements, which makes it easy to form a stable single-phase face-centered cubic (FCC) solid solution structure. By adjusting the proportion of elements or introducing alloying elements such as Al, Nb, and Ti, the wear resistance and corrosion resistance of its surface protective coating can be effectively optimized [7-9]. Specifically, an appropriate amount of Al doping can significantly enhance the tribological properties of the coating by promoting the formation of a dense oxide glaze layer [10]. However, excessive Al addition may lead to the precipitation of brittle phases, resulting in a significant deterioration of the material's plasticity [11]. The introduction of Nb element, through the solid solution strengthening mechanism and the Laves phase precipitation strengthening effect, not only enhances the strength of the alloy but also effectively maintains or even improves the ductility of the material due to its regulatory effect on dislocation movement [12, 13]. In addition, the addition of Ti element not only enhances the microhardness and wear resistance of the coating by promoting the precipitation of σ phase, but also inhibits crack propagation through the grain refinement effect, thereby optimizing the mechanical properties of the coating [14, 15]. Therefore, by precisely regulating the synergistic effect of multiple elements such as Al-Nb-Ti, a HEA coating system with excellent wear resistance, corrosion resistance and balanced mechanical properties can be constructed. This multi-scale collaborative optimization strategy provides a theoretical basis and technical path for the development of high-performance surface protection materials.

Among various surface modification technologies, laser cladding technology (LC) is an advanced manufacturing technique that uses a high-energy laser beam to melt metal powder and rapidly solidify it on the surface of the substrate. This technology has been widely applied in the field of material surface modification and damage repair due to its significant advantages such as a small heat-affected zone, low dilution rate, and high interfacial bonding strength [16, 17]. In the laser cladding process, the optimization of process parameters has a decisive influence on the quality of the cladding layer [18, 19]. Among them, the key process parameters include laser power, scanning speed and powder feeding rate, etc. The laser power directly determines the temperature field distribution and penetration depth of the molten pool, and thereby affects the dilution rate of the cladding layer and the interfacial bonding strength. The scanning speed significantly affects the microstructure evolution and mechanical properties of the cladding layer by regulating the cooling rate of the molten pool. The powder feeding rate needs to be coordinated and matched

with the laser power and scanning speed to ensure the uniformity and density of the cladding layer [20, 21]. By systematically optimizing the above-mentioned process parameters, the high densification and performance optimization of the cladding layer of CoCrFeNi series alloys can be achieved.

This study focuses on the regulatory mechanism of the addition of Al, Nb and Ti elements on the properties of CoCrFeNi series high-entropy alloys, and combines the optimization of laser cladding process parameters, aiming to develop high-performance alloy powders and enhance their mechanical properties. By exploring the synergistic mechanism of element addition and process parameters on alloy properties, and combining systematic experimental design and characterization analysis, this study will provide theoretical basis and technical support for the composition design and performance optimization of CoCrFeNi series high-entropy alloys.

2. Experimental procedure

2.1. Materials and Experiments

The base material selected for this study is 45# steel, with dimensions of 15mm×60mm×10mm. For specific chemical composition, please refer to Table 1. Before the experiment, the substrate surface was polished with SiC sandpaper (#400), and then ultrasonically cleaned with anhydrous ethanol to thoroughly remove surface oxides and other contaminants. The raw materials used in the experiment include CoCrFeNi pre-alloyed powder and high-purity (99.5%) Al, Nb and Ti metal powders (202401-PRD-31). In addition, when adding components, the lowest possible mixing enthalpy was considered to avoid the formation of intermetallic compounds, as shown in Figure 1. CoCrFeNiAl_xNb_yTi_z (x, y, z represent atomic percentages) alloy powder was prepared by mechanical alloying method. By regulating the addition amounts of Al, Nb and Ti, alloy powders with various compositions were prepared. The morphological characteristics of the powder and the laser cladding system are shown in Figure 2. The observation results of scanning electron microscopy (SEM) show that the particle size distribution range of the powder is 45-106 μm, with a high sphericity, indicating good fluidity. In addition, analysis by energy dispersive spectrometer (EDS) indicated that the spherical CoCrFeNi powder prepared by atomization had a uniform elemental distribution. Table 2 lists the detailed orthogonal proportioning schemes of the three powders.

Table 1. Chemical Composition of 45# Steel

Elements	C	Si	Mn	P	S	Mo	Cr	Cu	Fe
Content	0.45	0.23	0.64	0.012	0.01	0.003	0.075	0.01	Bal.

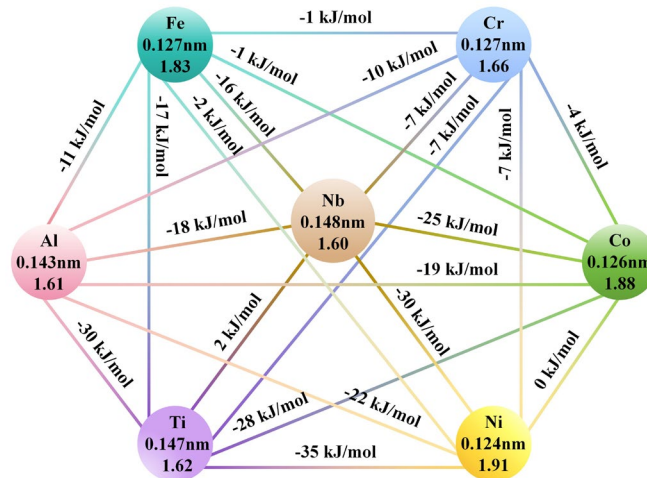


Figure 1. CoCrFeNiAl_xNb_yTi_z elements of atoms in the high entropy alloys coating system for mixing enthalpy, atomic radius and Pauling electronegativity

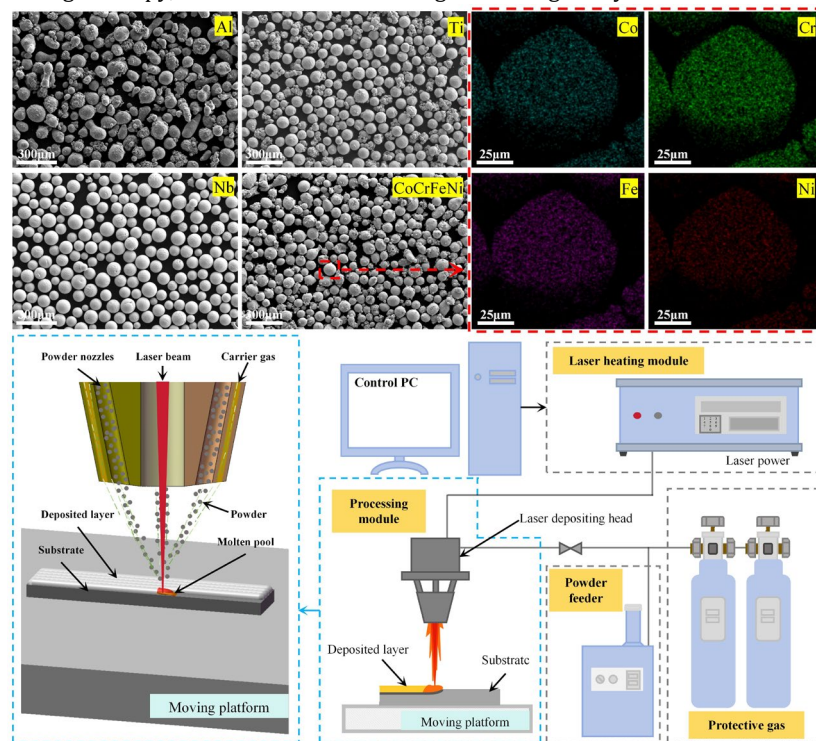


Figure 2. Powder and laser cladding system

Table 2. Chemical Composition of CoCrFeNiAl_xNb_yTi_z Powder (at.%)

Samples	HEAs	Co	Cr	Fe	Ni	Al	Nb	Ti
1	CoCrFeNi	25.00	25.00	25.00	25.00	-	-	-
2	CoCrFeNiNb _{0.3} Ti _{0.8}	19.61	19.61	19.61	19.61	-	5.88	15.68
3	CoCrFeNiNb _{0.3} Ti _{0.5}	20.83	20.83	20.83	20.83	-	6.25	10.43
4	CoCrFeNiAl _{0.7} Ti _{0.8}	18.18	18.18	18.18	18.18	12.73	-	14.55

5	CoCrFeNiAl _{0.7} Nb _{0.3} Ti _{0.5}	18.18	18.18	18.18	18.18	12.73	5.45	9.10
6	CoCrFeNiAl _{0.7} Nb _{0.5}	19.23	19.23	19.23	19.23	13.46	9.62	-
7	CoCrFeNiAlTi _{0.5}	18.18	18.18	18.18	18.18	18.18	-	9.10
8	CoCrFeNiAlNb _{0.3}	18.87	18.87	18.87	18.87	18.87	5.65	-
9	CoCrFeNiAlNb _{0.5} Ti _{0.8}	15.87	15.87	15.87	15.87	15.87	7.94	12.71

Figure 2 shows the laser cladding system, which is equipped with a three-way coaxial powder feeding device and can achieve precise powder transportation. During the experiment, the prepared powder was first precisely weighed, and then a planetary ball mill was used to ball grind it at a speed of 70 r/min for 1 hour to ensure uniform mixing of the powder. The powder after ball milling is placed in a drying oven and kept at 120°C for 2 hours to remove moisture and other volatile components. In addition, the entire laser cladding process is carried out in an argon-protected environment to prevent oxidation reactions during the cladding process. In the previous research, the appropriate range of process parameters was determined through a combination of experiments and numerical simulations. For each powder ratio, the L9(34) orthogonal test table was selected for the test. The orthogonal test arrangement is shown in Table 3. The experimental data of the surface morphology and crack imaging of the coating were analyzed by intuitive analysis method.

Table 3. Parameters of Laser Cladding Experiment

Sample		HEAs	Laser power(W)	Scanning rate(mm/s)	Feed rate(g/min)
1-9#	i ₁	CoCrFeNi	1000	3	9
	i ₂	CoCrFeNiNb _{0.3} Ti _{0.8}	1000	4	12
	i ₃	CoCrFeNiNb _{0.3} Ti _{0.5}	1000	5	15
	i ₄	CoCrFeNiAl _{0.7} Ti _{0.8}	1200	3	12
	i ₅	CoCrFeNiAl _{0.7} Nb _{0.3} Ti _{0.5}	1200	4	15
	i ₆	CoCrFeNiAl _{0.7} Nb _{0.5}	1200	5	9
	i ₇	CoCrFeNiAlTi _{0.5}	1400	3	15
	i ₈	CoCrFeNiAlNb _{0.3}	1400	4	9
	i ₉	CoCrFeNiAlNb _{0.5} Ti _{0.8}	1400	5	12

2.2. Characterization of coating structure

The macroscopic surface of the cladding coating was collected, and the number of cracks was counted using dye penetrant testing. Before conducting dye penetrant testing, the surface of the sample to be tested should be simply cleaned to ensure that there are no powder, rust, oil stains or other external factors that may affect dye penetrant testing on the surface of the sample to be tested. Then, dye penetrant testing was used for crack testing.

3. Conclusion and Discussion

The macroscopic morphology of the laser cladding layer directly affects the pro-

cessing efficiency and economy of industrial production. A superior cladding surface can reduce post-treatment processes and significantly enhance production efficiency. The existence of surface defects not only increases the post-processing procedures, but in severe cases, it may even lead to the scrapping of the workpiece, becoming the main bottleneck restricting the engineering application of this technology. Experimental studies [22-24] show that the morphology quality of the cladding layer is synergistically affected by process parameters such as laser power and scanning speed, as well as the thermal physical parameters of the cladding material. By optimizing the combination of process parameters, precise control of the morphology quality can be achieved. Figure 3 shows the surface morphology of the cladding layer formed by nine alloy composition ratios under different process parameters. It can be observed from the figure that, except for powder 6#, the other powders all exhibit good forming characteristics within the selected parameter range. The coating surface is generally smooth and continuous, without obvious molten pool ripples or accumulation defects, indicating a good match between the material and the process parameters. To further evaluate the compactness and crack sensitivity of the cladding layer, dye penetrant testing was used to detect cracks on the surfaces of each coating. As can be seen from Figure 4, no obvious penetrating cracks or large-area defects were found in coatings 1#, 2#, 3#, 4#, 5#, 7# and 8# during the inspection process. The number of pores and microcracks is relatively small, indicating that these ratios have good crack suppression ability and forming density under the current process parameters. In contrast, the surface of the 6# powder cladding layer has more irregular structures, and its macroscopic morphology is unstable. Combined with the subsequent crack imaging analysis, it may be related to improper alloy composition design or energy input matching. It is necessary to further optimize its parameter window or improve the powder formula to enhance its forming quality and structural integrity.

Alloy composition		1# CoCrFeNi	2# CoCrFeNi —Nb _{0.3} Ti _{0.8}	3# CoCrFeNi —Nb _{0.3} Ti _{0.5}	Scanning rate (mm/s)	Feed rate (g/min)
Macroscopic morphology	1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12
Alloy composition		4# CoCrFeNi —Al _{0.7} Ti _{0.8}	5# CoCrFeNi —Al _{0.7} Nb _{0.3} Ti _{0.5}	6# CoCrFeNi —Al _{0.7} Nb _{0.5}	Scanning rate (mm/s)	Feed rate (g/min)
Macroscopic morphology	1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12
Alloy composition		7# CoCrFeNi —AlTi _{0.5}	8# CoCrFeNi —AlNb _{0.3}	9# CoCrFeNi —AlNb _{0.5} Ti _{0.8}	Scanning rate (mm/s)	Feed rate (g/min)
Macroscopic morphology	1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12

Figure 3. Surface morphology of high-entropy alloy coating

Alloy composition		1# CoCrFeNi	2# CoCrFeNi —Nb _{0.3} Ti _{0.8}	3# CoCrFeNi —Nb _{0.3} Ti _{0.5}	Scanning rate (mm/s)	Feed rate (g/min)
Crack imaging	Laser power(W) 1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12
Alloy composition		4# CoCrFeNi —Al _{0.7} Ti _{0.8}	5# CoCrFeNi —Al _{0.7} Nb _{0.3} Ti _{0.5}	6# CoCrFeNi —Al _{0.7} Nb _{0.5}	Scanning rate (mm/s)	Feed rate (g/min)
Crack imaging	Laser power(W) 1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12
Alloy composition		7# CoCrFeNi —AlTi _{0.5}	8# CoCrFeNi —AlNb _{0.3}	9# CoCrFeNi —AlNb _{0.5} Ti _{0.8}	Scanning rate (mm/s)	Feed rate (g/min)
Crack imaging	Laser power(W) 1000				3	9
					4	12
					5	15
	1200				3	12
					4	15
					5	9
	1400				3	15
					4	9
					5	12

Figure 4. Crack imaging of high-entropy alloy coating

3.1. Summary

- In this study, the CoCrFeNiAlxNbyTiz multi-element high-entropy alloy system was constructed through orthogonal experimental design, and the laser cladding process parameters were optimized. Based on the observation of the surface morphology of the coating and the analysis of crack imaging, the designed alloy system has a good match with the process parameters, and a high-entropy alloy composition system with better surface formation quality can be selected.

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