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Phase assemblage and wear resistance of laser-cladding Al$_{0.8}$FeCoNiCrCu$_{0.5}$Si$_x$ high-entropy alloys on aluminum

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Abstract

Preparing a coating with excellent mechanical properties on aluminum substrates by laser cladding has always been challenging because of the low melting point and high chemical activity of Al. In this study, we designed Al$_{0.8}$FeCoNiCrCu$_{0.5}$Si$_x$ (x = 0, 0.2, 0.3, 0.4, and 0.5) high-entropy alloys (HEAs) as cladding materials. The proposed study aims to exploit the unique high-entropy effect of HEAs to restrict the formation of hard and brittle intermetallic compounds via the reaction between Al in the substrate and added powders, thereby enhancing the quality of the formed coatings and ultimately improving the surface properties of the Al alloy. Results show that with an increase in the Si content, the structure of the Al$_{0.8}$CrFeCoNiCu$_{0.5}$Si$_x$ coating changes from FCC + BCC1 + BCC2 to BCC1 + BCC2. The hardness of the Al$_{0.8}$FeCoNiCrCu$_{0.5}$Si$_x$ coating first increases and then decreases with an increase in the Si content. The coatings with the highest and lowest hardness were those with compositions of Al$_{0.8}$FeCoNiCrCu$_{0.5}$Si$_{0.4}$ (592 HV$_{0.2}$) and Al$_{0.8}$FeCoNiCrCu$_{0.5}$ (412 HV$_{0.2}$), respectively, which is approximately seven and five times greater than that of the substrate, respectively. The effect of Si content on the wear resistance of the coating is the same as its effect on the hardness. The wear rates of coatings with different Si contents range from $1.19 	imes 10^{-6}$ to $8.99 	imes 10^{-7}$ mm$^3$ Nm$^{-1}$ and are only 0.34% to 0.25% of the substrate. Obvious, the Al$_{0.8}$FeCoNiCrCu$_{0.5}$Si$_x$ HEAs can be used as coating materials to improve the mechanical properties of an Al alloy surface.

1. Introduction

Aluminum (Al) alloy is the preferred material for mechanical lightweight design owing to its low density, high specific strength, and easy availability [1–4]. However, the low hardness and poor wear of the Al alloy surface limit its application, especially in components with stringent reliability requirements. In recent years, surface treatment methods, such as electroplating, thermal spraying, and laser cladding (LC), have been used to enhance the surface properties of Al alloys [5–8]. Among them, LC generates less pollution, has a high work efficiency, and can be used to apply thick coatings; therefore, it has distinct advantages over other surface treatment methods [9–13]. However, cladding materials with good mechanical properties, such as Fe- and Ni-based powders and ceramic materials, usually have a high melting point. In the LC process, to ensure the full melting of the cladding materials, the laser is operated at a high output power. Because the melting point of Al alloy is low, Al in the substrate will inevitably float to the cladding layer. Because of the large electronegativity of Al, fragile intermetallic compounds with a complex phase structure are easily formed after reaction between Al in the substrate and aforementioned coating materials, resulting in an increase in cracking sensitivity of the coating [14–17]. Therefore, applying an LC coating to Al has always been challenging in the manufacturing field.

Since their development in 2004, high-entropy alloys (HEAs) have become a promising material system with high academic research value and strong industrial development potential [18]. HEAs comprised five or more
metal elements in an isatomic or approximately isatomic ratio and tend to form simple solid solutions rather than intermetallic compounds with complex phase structures [19–21]. HEAs have high hardness, high plasticity, and good oxidation resistance, among other excellent properties, and tend to form simple solid solutions rather than intermetallic compounds with complex phase structures [22–24], which makes it a potential application as a protective coating. Guo et al investigated the influence of Ti and C addition on the phase structure and comprehensive properties of CoCrCuFeNiSi0.3 HEAs coating on 304 stainless steels by laser cladding [25]. The CoCrCuFeNiSi0.3 HEAs coating was composed of FCC solid solution structure, by the addition of Ti and C, the coatings consist of FCC solid solution and TiC, and average microhardness and wear volume of CoCrCuFeNiSi0.3 HEA coating is 498.5 HV0.2 and 0.42 mm3, respectively. Meng et al [26] prepared the AlCrFeCoNiCu HEA particles on the surface of AZ91D magnesium alloy by laser casting. Although the reinforcing phase particles and the Mg matrix coexist in the molten state, no intermetallic compound with complex phase structure was formed in the coating after solidification due to high mixing entropy of the alloy. Previous research has shown that Al exhibits high solid solubility in the HEA system comprising Fe, Co, Ni, Cr, and Cu, which means that the Al in the substrate and the added powder will tend to form solid solutions after reaction, thereby reducing the susceptibility of the coating to cracking [27–29]. In addition, Si is a more abundant element in nature, compared with Al, its atomic radius is smaller, and its mixing enthalpy with metal elements is more negative. The addition of Si will increase the mixed entropy and the degree of lattice distortion of HEA systems and avoid the consumption of more precious metals [30].

In this paper, Al0.8FeCoNiCrCu0.5Si0.5 HEA coatings with different Si contents were prepared on the surface of Al alloy by coaxial LC. The phase structure, microstructure, hardness, and wear resistance of the coatings were studied with the aim of identifying the best coating composition for the surface modification of Al materials.

2. Materials and methods

5083 Al alloy was selected as the substrate. A sample with dimensions of 50 × 30 × 10 mm3 was used because of its good superplasticity and weldability. Powdered Al, Fe, Co, Ni, Cr, Cu, and Si with 99.9% purity were used, all with a particle size of 200–325 mesh. Used ball mill to fully mix the powder according to moral ratio of Al0.8FeCoNiCrCu0.5Si0.5 (x = 0, 0.2, 0.3, 0.4, 0.5).

The CO2 laser was a ROFIN model DC050. The powder feeding device was used, and the shielding gas was argon. Based on previous experiments [29, 31] the process parameters are as follows: spot diameter 1.2 mm; laser power 1850 W, powder feed rate 5.6 g min−1, scanning speed 120 mm min−1, duty cycle 70%, pulse frequency 50 Hz, overlap rate 30%, and carrier gas flow rate 5 l min−1. The powder ratio and alloy abbreviations are shown in table 1.

The phase of the coating sample was analyzed by Empyrean sharp x-ray diffraction (XRD) with Cu Kα radiation (λ = 1.5406 Å), at a scanning speed of 4 deg min−1 and in the angle scan range of 20°–90°. The middle section of the coating was cut perpendicular along the scanning direction. It was then burnished with 400# sandpaper and was polished with 2 μm alumina suspension. The HEA coating was etched with the mixed solution of hydrochloric acid and nitric acid (volume ratio 3:1). The microstructure of the coating was observed on a scanning electron microscope (SEM, JSM-6510F), and the composition was analyzed by an energy dispersive spectrometer (EDS). Transmission electron microscope (TEM, JEM-2100F) was used to further analyze the phase structure of the sample. The 0.5 mm thickness sample was prepared by wire cutting and was polished to 50–80 μm by a sandpaper. It was then made into a thin circle with 3 mm diameter by a punching mechanism. Then, the sample was thinned by electrolysis in 5% perchlorate alcohol environment until perforation. Finally, it was reduced to 30–200 nm by the plasma thinning machine. The hardness of the coating was measured by a microhardness tester (MH-60), where the input load was 200 g, and the loading time was 10 s.

The wear test was performed at room temperature. The wear ring used a GCr15 steel ring with a hardness of 61 HRC, a diameter of 50 mm, and a thickness of 10 mm with a wear sample size of 7 × 14 × 10 mm. The test load was 98 N, the speed was 400 r min−1, and time allowed was 30 min. The wear rate of the material was evaluated by calculating the wear volume. The formula is as follows:

Table 1. Nominal components and abbreviations of Al0.8FeCoNiCrCu0.5Six coatings (at%).

| Alloy Abbreviation | Al   | Cr   | Fe   | Co   | Ni   | Si   | Cu   |
|-------------------|------|------|------|------|------|------|------|
| Si0               | 15.09| 18.86| 18.86| 18.86| 0    | 9.43 |
| Si0.2             | 14.54| 18.18| 18.18| 18.18| 3.63 | 9.09 |
| Si0.3             | 14.28| 17.85| 17.85| 17.85| 5.35 | 8.92 |
| Si0.4             | 14.03| 17.54| 17.54| 17.54| 7.01 | 8.77 |
| Si0.5             | 13.79| 17.24| 17.24| 17.24| 8.62 | 8.62 |
\[ w = \frac{V_{\text{loss}}}{L \times N} \]  

\[ L = 2\pi R v t \]  

\[ V_{\text{loss}} = B \left[ \frac{\pi R^2}{180} \arcsin \left( \frac{b}{2R} \right) - \frac{b}{2} \sqrt{R^2 - \frac{b^2}{4}} \right] \]

where \( w \) is the wear rate, \( V_{\text{loss}} \) is the wear volume (mm\(^3\)), \( B \) is the wear track length (mm), \( b \) is the wear track width (mm), \( v \) is the rotating speed of the wear ring (r min\(^{-1}\)), and \( L \) is the total wear ring sliding length (mm).

Figure 1 shows a schematic of the wear test.

Figure 2. XRD patterns of the Al\(_{0.8}\)FeCoNiCrCu\(_{0.5}\)Si\(_x\) coatings.

3. Results and discussion

3.1. Phase and microstructure

Figure 2 shows the x-ray diffraction (XRD) pattern of the Al\(_{0.8}\)CrFeCoNiCu\(_{0.5}\)Si\(_x\) coatings. When \( 0 \leq x \leq 0.3 \), the coating includes BCC1 and BCC2 phases and an FCC phase. The BCC1 phase is a disordered BCC solid solution with three peaks at approximately \( 2\theta = 44.5^\circ, 64.0^\circ, \) and \( 82^\circ \), similar to the \( \alpha-(\text{Fe,Cr}) \) phase (PDF\#003-1050 for Fe, PDF\#001-1250 for Cr). The (100) peak at \( 2\theta = 31^\circ \) confirms that the BCC2 phase is an ordered BCC phase, corresponding to the AlNi phase (PDF\#002-1261). The peaks of the FCC phase at \( 2\theta = 43.5^\circ, 51^\circ, \) and \( 74.5^\circ \) are similar to those of \( \alpha-\text{Cu} \) (PDF\#001-1241). Further clarification will be provided by TEM analysis. When \( x > 0.3 \), the peak of the FCC phase disappeared and the intensity of the main BCC peak at \( 2\theta = 44.5^\circ \) increased, indicating that the addition of Si promoted the phase transformation from the FCC...
phase to the BCC phase. This transformation is attributed mainly to the large difference between the atomic radius of Si and the other elements in the alloy; the added Si increases the degree of lattice distortion of the FCC phase, thereby increasing the lattice strain energy. As the Si content increases further, the excessive lattice strain energy renders the FCC phase with a high packing density unable to maintain a stable structure. To release the lattice strain energy, the FCC phase transitions to the BCC phase with a low packing density. Kumar et al and Liu et al also found that Si promotes the formation of BCC phases in AlFeCoNiCuCrSix and Al0.5FeCoNiCrCuSix HEAs. Unlike traditional alloys, various elements are mixed to form a simple phase structure instead of a large number of complex intermetallic compounds, mainly because of the high entropy effect of HEAs.

Notably, the positions of peaks of the solid solution in the coatings prepared in the present work are not strictly consistent with the standard peaks in the PDF cards, mainly because the presence of more elements in the coatings changes the lattice constant of the solid solution.

Figure 3 shows the macro morphology and microstructure of Si0 coating (i.e., Al0.8CrFeCoNiCu0.5Six with x = 0). Table 2 shows the EDS results for each region of the coatings. Figure 3(a) shows a macro and a cross-section image of the Si0 coating. No obvious defects such as pores or cracks are observed in the cladding layer. The thickness of the coating is approximately 662 ± 74 μm. Figure 3(b) shows an enlarged view of the coating interface. The microstructure of the heat-affected zone is clearly observed after the sample was slightly corroded by hydrofluoric acid; however, the microstructure of the cladding was not visible and the sample required treatment with aqua regia for 2 ~ 3 min. Figure 3(c) shows the microstructure in the middle of the Si0 coating, where dendrite (DR) and interdendrite (ID) structure are observed. The EDS results show that Fe and Cr are enriched in the dendritic region, and they are the main constituent elements of BCC1 phase. At the same time, the area of the dendritic region is much higher than that of the interdendritic region, and the diffraction peak intensity of XRD of BCC phase is much higher than that of FCC phase. Therefore, the dendritic region may be BCC structure. In the same way, the diffraction peak intensity of XRD of Cu-rich FCC phase is lower than that of BCC phase, the area of interdendritic region is much smaller than that of the dendritic region and Cu is enriched in the interdendritic region, so it is speculated that the interdendritic region is FCC phase. To further verify the phase of each structure region, the Si6 coating was analyzed by transmission electron microscopy (TEM) and selected-area electron diffraction (SAED).
Figure 4 shows bright-field TEM images of the Si0 coating. Some elliptic precipitates are distributed on the matrix. The \([001]\) zone axis diffraction pattern confirms that the precipitates are an ordered BCC phase and that the matrix is a disordered BCC phase (figures 4(b) and (c), respectively). Also, the interdendritic region is identified as FCC on the basis of the \([100]\) zone axis diffraction (figure 4(d)). Both the matrix and the precipitate have the same crystal structures but different chemical compositions; they exhibit only a small lattice misfit and a specific crystallographic coherence. Thus, the precipitates exhibit an elliptic shape [34].

When the Si content is 0.2, the coating shows an equiaxed dendrite structure, as shown in figures 5(a) and (b). By contrast, when the Si content reaches 0.3, a chrysanthemum dendrite morphology is observed, as shown in figures 5(c) and (d). When the Si content is 0.4, the dendritic morphology changes to a network structure, and the dendrite region is Fe- and Cr-rich BCC1 phase and the interdendrites are Al- and Ni-rich B2 phase, as shown in figures 5(e) and (h). The TEM analysis shown in figure 6. Unlike the Si0 coating (figure 4), the Si0.4 coating contains no ellipsoidal particles. The SAED shows that the Si0.4 coating only consist of BCC phase (figures 6(a) and (b)). Meanwhile, a high dislocation density was identified from the HRTEM image and the corresponding SAED pattern, as shown in figures 6(d) and (e), respectively. As shown in figures 5(g) and (h), the Si0.4 coating shows no reticular dendrite structure; however, a secondary dendrite arm emerges. The EDS results show that the element distribution in the dendrite and interdendrite of the Si0.4 coating is similar to that of the Si0.4 coating; the dendrite rich in Fe and Cr has the BCC1 structure, and the interdendrite rich in Al and Ni has the BCC2 structure. It is worth mentioning that, considering the evaporation effect of Al, the content of Al in each

| Areas  | Al   | Cr  | Fe  | Co  | Ni  | Cu  | Si  |
|-------|------|-----|-----|-----|-----|-----|-----|
| Si0   | 15.09| 18.86| 18.86| 18.86| 18.86| 9.43| 0   |
| DR1   | 18.23| 18.23| 19.71| 20.95| 19.84| 15.59| 0   |
| ID1   | 20.42| 10.36| 12.75|12.25 | 20.8 | 23.42| 0   |
| Si0.2 | 14.54| 18.18| 18.18| 18.18| 18.18| 9.09 |3.63 |
| DR2   | 13.62| 20.52|21.98 |19.95 |14.42 | 5.54 |3.97 |
| ID2   | 19.42| 10.95| 12.25| 18.16| 20.84| 15.34| 3.04|
| Si0.3 | 14.29| 17.86|17.86 |17.86 |17.86 | 8.93 |5.36 |
| DR3   | 13.45| 21.18|22.52 |14.75 |13.05 | 8.53 |6.52 |
| ID3   | 19.53| 11.74|10.84 |20.10 |21.97 |10.86 |4.96 |
| Si0.4 | 14.04| 17.55|17.55 |17.55 |17.55 | 8.77 |7.01 |
| DR4   | 13.31| 19.85|22.08 |16.91 |10.85 | 8.42 |8.58 |
| ID4   | 20.98| 9.75 |10.32 |17.95 |24.24 | 9.74 |7.02 |
| Si0.5 | 13.79| 17.24|17.24 |17.24 |17.24 | 8.62 |8.62 |
| DR5   | 11.45| 20.47|21.45 |16.57 |12.98 | 7.96 |9.12 |
| ID5   | 19.74| 8.45 | 9.5 |17.42 |26.42 | 9.52 |8.95 |
coating is still close to or higher than the nominal composition, indicating Al in substrate floats up to the coating during LC. But the intermetallic compound with complex phase structure have not been detected, which indicates the coating defects caused by the dilution behavior of the substrate is controlled to some degree by process parameters and power ratio in this experiment.

3.2. Hardness and wear tests

Figure 7 shows the average hardness and wear-rate curves of the Al<sub>0.8</sub>CrFeCoNiCu<sub>0.5</sub>Si<sub>x</sub> coatings with different Si contents and substrates. Table 3 shows the calculation results for the wear rates and average hardness of coatings and substrates, where the wear rate was calculated on the basis of equations (1)–(3). With increasing Si content, the hardness of the coatings increases first and then decreases. The phase structure of the Si<sub>0</sub>, Si<sub>0.2</sub>, and Si<sub>0.3</sub> coating is the same; however, the hardness is improved with increasing Si content. This result is mainly attributed to the substantial difference in atomic radius between Si with the other elements, which results in an increase in the degree of lattice distortion. The increase of the volume fraction of the BCC phase in the Si<sub>0.4</sub> is responsible for the further improvement of the hardness with increasing Si content. However, when the Si content was increased to 0.5, the fine network dendrite disappeared and coarse dendrite was formed, resulting in a decrease in hardness. With increasing Si content, the wear rate of the Al<sub>0.8</sub>CrFeCoNiCu<sub>0.5</sub>Si<sub>x</sub> HEAs first decreases; when the Si content reaches 0.4, the minimum value is reached, which is the opposite trend observed for the hardness, which in line with the Archard equation [35]. Lower wear rate means better wear resistance, so Si<sub>0.4</sub> shows the best wear resistance. To analyze the wear mechanism of each sample, we observed their wear surfaces by SEM.

Figure 8 shows the surface morphology of the Al<sub>0.8</sub>CrFeCoNiCu<sub>0.5</sub>Si<sub>x</sub> coatings and substrates after wear experiments. The HEA coatings exhibit a smoother wear surface overall relative to the substrate. As shown in figure 8(a), severe plastic deformation along the wear direction, elongated dimples, and a stepped fracture were observed on the Al alloy wear surface, indicating that serious adhesion wear and delamination fracture occurred during the wear test. Figure 8(b) shows that a peeling surface, furrow, and fine abrasive particles appear on the wear surface of the Si10 coating. The wear mechanism is adhesive wear and abrasive wear. Similarly, the wear
The surface of the Si0.2 coating shows a peeling surface and furrow, and the wear mechanism is the same as that of the Si0 coating, as shown in Figure 8 (c). When the Si content is greater than 0.2, the peeling surface disappears and a large number of abrasive particles appear; the wear mechanism of the Si0.3 coating becomes abrasive wear, as shown in Figure 8 (d). Si0.4 coating is also abrasive wear, but the abrasive particles are reduced and the furrow is shallower, indicating that it has better wear resistance than Si0.3 coating, as shown in Figure 8 (e).

Although the hardness of Si0.3 coating and Si0.4 coating are similar, the wear rate of Si0.4 coating is lower, the reason may be speculated as follows: The XRD results show that there are fewer FCC phase in the Si0.4 coating, and the wear morphology shows that the number of wear particles of the Si0.4 coating are less than that of the Si0.3 coating, it can be estimated that the less FCC phase as the abrasive particles reduces the surface wear degree in during the wear process, so the wear rate of the Si0.4 coating is lower.
When the Si content reaches \( x = 0.5 \), the adhesion layer and furrow interval distribute on the wear surface, as shown in figure 8(f). A comparison of the wear resistances of the Al_{0.8}CrFeCoNiCu_{0.5}Si_x coatings with different Si contents indicates that increasing Si content improves the coating wear resistance except for the Si_{0.5}. The greater volume fraction of BCC phase of the coatings enhances resistance to microcutting during the wear process, which in turn reduces plastic deformation during the wear process. Therefore, the wear surface of the Si_{0.3} and Si_{0.4} coatings with less FCC phase and more BCC phase is smoother than that of Si_0, Si_{0.2}, and Si_{0.3} coatings. In particular, although the phase structure of the Si_{0.5} coating is the same as that of the Si_{0.4} coating, the hardness of the Si_{0.5} coating is lower because of the formation of a coarse microstructure, which leads to diminished ability of the microconvex body to resist the deformation force; consequently, the wear degree of the Si_{0.5} coating is higher than that of the Si_{0.4} coating.

4. Conclusion

The microstructure and properties of LC Al_{0.8}FeCoNiCrCu_{0.5}Si_x HEA coatings on Al were studied. The main conclusions are summarized as follows:

1. The Al_{0.8}CrFeCoNiCu_{0.5}Si_x coatings have a simple BCC and FCC structure; no intermetallic compound with a complex phase structure was observed. With increasing Si content, the proportion of the FCC phase decreased and that of the BCC phase increased. When the Si content was increased to 0.4, the FCC phase disappeared and the Al_{0.8}CrFeCoNiCu_{0.5}Si_{0.4} coating comprised only BCC1 and BCC2 phases.

2. With increasing Si content, the hardness of the Al_{0.8}FeCoNiCrCu_{0.5}Si_x coatings first increases and then decreases. The average hardness of the Si_0, Si_{0.2}, Si_{0.3}, Si_{0.4}, and Si_{0.5} was 479 HV_{0.2}, 536 HV_{0.2}, 587 HV_{0.2}, 592 HV_{0.2}, and 573 HV_{0.2}, respectively, which are five to seven times greater than the hardness of the substrate.

3. The wear resistance of the Al_{0.8}FeCoNiCrCu_{0.5}Si_x coatings first increases and then decreases with increasing Si content. The wear rate of the Al_{0.8}CrFeCoNiCu_{0.5}Si_{0.4}, which exhibited the smallest wear rate among the investigated coatings, was \( 2.63 \times 10^{-7} \text{ mm}^3 \text{ Nm}^{-1} \), which is only 0.07% of the wear rate of the substrate. The wear rate of the Al_{0.8}FeCoNiCrCu_{0.5} coating with the highest wear rate was \( 1.63 \times 10^{-6} \text{ mm}^3 \text{ Nm}^{-1} \), which is 0.34% of the wear rate of the substrate. Obviously, the Al_{0.8}FeCoNiCrCu_{0.5}Si_x HEAs can be used as coating materials to improve the mechanical properties of Al alloy surfaces.

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