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Microstructure and properties of high-entropy alloy reinforced aluminum matrix composites by spark plasma sintering



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ABSTRACT

An CoCrFeMnNi high-entropy alloy (HEA) particles reinforced 2024Al alloy composite was prepared by spark plasma sintering (SPS). There is an interdiffusion (ID) layer between the HEA particle and the matrix. The composition and micromechanical properties of the diffusion layer were investigated by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), electron probe and nanoindentation. The unique element distribution in the diffusion layer and the obvious creep phenomenon are related to the sintering mode. The hardness of the composite is increased 63.7%, which was attributed to particle reinforcement and the relative low content of sintering defects in the matrix.

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1. Introduction

Metal matrix composites (MMCs) are metal/alloy materials with ceramic reinforcement. It is known for its high specific strength, modulus and hardness [1,2]. They account for a large proportion of aerospace applications [3–6]. Ceramic particles are the most widely used as reinforcement in traditional metal matrix composites, such as SiC, Al_2O_3 , TiB₂, etc. However, The thermal expansion coefficient mismatch between ceramic particles and matrix, ceramic particle easy to fragmentation and poor interface bonding, result in poor plasticity and toughness of composites [7]. To overcome the weakness of metal-ceramic composites, metal-metal composite systems is a better choice due to the natural good interface bonding. Firstly, metallic glasses (such as zirconium base, aluminum base, magnesium base, etc.) were used as reinforcing particles to strengthen metals. However, metallic glasses also have high brittleness and low crystallization temperature [8,9], which

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makes them difficult to obtain higher sintering quality in low-temperature sintering process [10].

Different from the traditionally multi-component alloys, high entropy alloys (HEAs) are defined as alloys containing at least five major elements with a concentration range between 5 and 35 at.%, with simple solid solution structures [11]. Moreover, HEAs also have many promising properties, such as high strength, good wear resistance and outstanding thermal stability [12–14]. Especially, high entropy alloys have higher elongation compared with ceramics and metal-glasses. Therefore, high entropy alloys are a very good candidate as reinforcement to prepare metal matrix composites.

Some work has already been carried out using HEAs reinforcements in metal matrix composites. Wang et al. used FeNiCrCoAl₃ particles to reinforce 2024 aluminum matrix composites, whose compressive strength was 710 MPa [15]. Meng et al. prepared AlCoCrCuFeNi particle reinforced AZ91D matrix composite coating by laser surface forming. High entropy alloy reacted with magnesium alloy matrix to form new phase, and the wear resistance of the coating was greatly improved [16,17]. Chen et al. prepared AlCoNiCrFe high entropy alloy reinforced Cu matrix

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composites by powder metallurgy. The yield strength of composites increased more than 160% compared with the matrix, and the elongation reached to 15%. Liu et al. studied the effect of transition layer on AlCoCrFeNi high-entropy alloy paticles reinforced aluminum matrix composites. With the help of transmission electron microscope, it was found that the interface layer is FCC structure [9].

In this paper, CoCrFeMnNi high-entropy alloy particles were used as reinforcement, and 2024 aluminum powder was used as matrix. The aluminum matrix composites were prepared by spark plasma sintering method. During the sintering process, a diffusion layer was formed between the CoCrFeMnNi HEA particles and the Al matrix. The microstructure and mechanical properties of composites were studied.

2. Experimental

2.1. Samples preparation

A commercial 2024 Al alloy powder was used as matrix material, with high sphericity and smooth surface. The particles size was $45-105 \,\mu$ m. The chemical composition (wt.%) of 2024Al was 3.83 Cu, 1.57 Mg, 0.61 Mn, 0.23 Zn, 0.12 Cr and balance with Al. Single-phase CoCrFeMnNi high-entropy alloy powder particle size <50 μ m. The high-entropy alloy powder was added to the aluminum powder, and the mixture is uniformly mixed by low-energy ball milling in order to avoid deformation of powder. To prevent the strong agglomeration of the powder, ball milling in a vacuum environment. Given the strength and density of the composite, the volume fraction of the HEA powder was determined to be 7%.

The mixed powder was consolidated by spark plasma sintering at 823K and 40 MPa with vacuum <10Pa. Fig. 1 was shown the process route for spark plasma sintering. A sample with size of 20 mm \times 10 mm was obtained after sintering. The microstructure was examined using scanning electron microscopy (SEM) equipped with energy dispersive spectrometer (EDS), and X-Ray diffraction (XRD). The diffusion layer was quantitatively analyzed by electron probe.

2.2. Nanoindentation experiment

The nanoindentation was performed on the sintered composites, using Hysitron-Picoindenter with Berkovich diamond tip at room temperature. The indentation test was performed from the particles through the interface to the matrix direction, and the



Fig. 1. Temperature and pressure vs. time during SPS process.

indentation load was set to 800 μ N. To avoid the influence of indentation result by the adjacent indentation, and ensure the accuracy and reliability of the experimental results, there were 13 indentation test points with a fixed pitch of 1 μ m. Before nano-indentation test, the alloy was mechanically polished to a mirror finish.

3. Results and discussions

3.1. Microstructure

Fig. 2 shows the XRD patterns of the sintered composite. In addition to matrix Al phase and a small amount of CoCrFeMnNi phase, new AlCu₂Mn phase and MnAl₆ phase are formed in the sintered products of bulk composites. The results show that during the heating and sintering process, Cu and Mn atoms were precipitated from the solid solution, and the precipitated Cu and Mn atoms react with the surrounding Al atoms to form a new phase under the activation of the discharge plasma. This indicates that the reinforcement and the matrix have a certain degree of interfacial reaction, resulting in an interface phase.

It is shown the SEM image of CoCrFeMnNi HEA powder in Fig. 3. It can be seen that they have an approximately spherical shape with a smooth surface. Fig. 4 is SEM image of the 7 vol% CoCrFeMnNi HEA particles reinforced aluminum matrix composite. As shown in Fig. 4a, the microstructure is consisted of a continuous gray matrix phase and bright white particle phase. The spherical particles with different sizes are evenly distributed in the matrix to form a typical composite structure. Compared with the high entropy particles in Fig. 3, the HEA particles did not undergo severe deformation during ball milling. It can be seen that there was an obvious diffusion (ID) layer between the HEA particles and the matrix in the composite (see Fig. 4b). The diffusion layer exhibited a different color, and the inner diffusion layer near the high entropy alloy particles is gray and the shape was close to a ring shape. The outer diffusion layer is lighter in color than the inner diffusion layer, and the shape is irregular. The diffusion layer is relatively flat on the side in contact with the high-entropy alloy, but is serrated on the side in contact with aluminum matrix. The reason for this is mainly due to the uneven diffusion of elements. Atoms can jump randomly in all directions during the diffusion process. This will result in a concentration difference between the same elements at different locations on the interface, and the solid-liquid interface reaction will not



Fig. 2. XRD patterns of the composite.



Fig. 3. SEM images of the HEA particles: (a) low magnification; (b) high magnification.



Fig. 4. SEM images of the composite: (a) low magnification; (b) high magnification.

occur simultaneously (More detail in the following section). It can only be that the high concentration region of the element on the interface reacts first, and then grows rapidly, which is the reason why the serrated shape will appear along the side of the aluminum matrix. The prepared HEAp/Al structure is dense, and the surface of the sample has no obvious pores, which indicates that the reinforcement and the matrix can effectively wet. The ID layer with a thickness of 6 μ m, this is consistent with the results reported by Liu et al. [9].

3.2. Diffusion layer structure

Fig. 5 shows the EDS result (line scanning) corresponding to the green line in the figure, which shows the distribution of elements in the HEA particle, ID layer, and matrix. The inset image is an

enlarged view of the red box selection area. The content of the element is characterized by the magnitude of the *k* factor. The calculation of the *k* factor is determined according to the ratio standard, so when the *k* factor fluctuates less, the intensity of the element can be ignored. In Fig. 5, it is clear that when the line scanning distance is less than 9.5 μ m (in the matrix), the elements are primarily Al, Cu and Mg. At this time, the *k* factor of the other elements is less than 10, and it is considered that these elements are not existing in the matrix. According to the line scanning, it can clearly observe that the distribution of Mn, Cr, Fe, Co and Ni elements from the reinforcement to the matrix is approximately the same. Moreover, the element content is slowly reduced, and the content of Al is significantly increased. Compared with the diffusion of Cu, the diffusion rate of each component of the high-entropy alloy in the ID layer is relatively small. Because of the difference



Fig. 5. EDS results (line scanning) of the green line region in composite. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

of high concentration of elements between HEA particles and matrix powder, the elements may diffuse to each other near the interface during sintering. Cu atoms segregate at the diffusion layer because the mixing entropy between Cu and Co, Cr, Fe, Ni, Mn is not enough to balance the enthalpy of mixing between them, so that Cu atoms do not dissolve well with other atoms ostracized to the grain boundary.

In order to study the interface bonding between the reinforcement and the matrix, the compositional changes of the diffusion layer were tested in detail. Fig. 6 is the element distribution diagram of the high-entropy alloy reinforced Al matrix composite. The Mn, Fe, Cr, Co and Ni elements are evenly distributed in the diffusion layer. The chemical composition of the spots marked in Fig. 6 was analyzed and the results are given in Table 1. It can be seen that the element distribution of high entropy alloy does not accord with the typical diffusion kinetics, which is related to the sintering mode. Compared with the traditional hot-pressing sintering method, the densification of SPS depends on the flow of current between metal particles. Liu [18] and Zhao [19] established a sintering model when investigating the temperature distribution and neck formation of combined particles during SPS. According to Tan's theory [20], in the process of SPS, a local high temperature higher than the melting point of matrix powder was produced around the high entropy alloy particles. Therefore, the formation of ID layer cannot be attributed only to the solid/solid interaction diffusion between HEA particles and matrix powder. It is reported that HEA has a higher resistivity than conventional alloys, so the strong loule thermal effect produced by pulse current does not make the regional temperature higher than the melting point of HEA. Due to the different diffusion coefficients of high entropy alloying elements in liquid aluminum and solid aluminum, the mutual diffusion layer has obvious delamination (as shown in Fig. 4). In the melting zone, since the Al and Cu atoms are too large, and the melting point and cohesive energy of the high-entropy alloy are relatively high, it is difficult for Al and Cu atoms to diffuse into the solid high-entropy alloy to form a new solid solution, which is consistent with the XRD results. Since Fe, Ni and Co elements have a large diffusion coefficient with respect to other

Table 1

Chemical composition of diffusion layer of CoCrFeMnNi HEA particles reinforced Al matrix composites (at.%).

Regions	Al	Fe	Mn	Cu	Cr	Ni	Со	Mg
А	67.029	3.035	3.451	16.560	3.457	3.404	2.691	0.374
В	80.198	4.539	1.650	3.351	0.774	4.111	5.084	0.293
С	83.757	3.729	1.245	2.903	0.526	3.584	3.74	0.516

elements in aluminum, Fe, Ni and Co cannot be precipitated in the aluminum melt far from the reaction layer during rapid solidification. They are formed the rich area of Fe, Ni and Co at the boundary of the inner diffusion layer. The distribution of elements in the outer diffusion layer should be consistent with the distribution at the boundary, which is consistent with the results.

3.3. Micromechanical properties

Fig. 7 is shown the image of the indented particles under the Hysitron-Picoindenter. The indentation points were closely arranged at a pitch of 1 μ m and pass through the interface in the direction of the arrow. The shape of the indentation can be clearly seen from the figure. As the indentation position moves from the particle to the matrix, the protrusion formed by the indentation gradually becomes larger. At the interface, the image shows a significant build-up around the dent, indicating a highly localized plastic deformation under the nanoindentation. It can be seen that the diffusion layer has a large roughness, which may affect the result of the indentation process. However, it can be seen from the *p*-*h* curve does not fluctuate greatly in Fig. 8, and at the same time, as the depth of the indentation increases, the effect of roughness on the results becomes smaller and smaller.

Oliver and Pharr developed a comprehensive method for determining depth sensing indentation hardness and elastic modulus using load-displacement data [21]. For the Berkovich diamond indenter, the projected contact area A_c can be achieved by contact indentation depth h_c and regional function.



Fig. 6. Electron probe results of different elements in the Al matrix composite.



Fig. 7. The image of indentation in composite under the Hysitron-Picoindenter.



Fig. 8. The P-h curves of different micro structures in MMC.

$$A_{C} = C_{0}h_{c}^{2} + C_{1}h_{c} + C_{2}h_{c}^{1/2} + C_{3}h_{c}^{1/4} + C_{4}h_{c}^{1/8}$$
(1)

Where C_0 , C_1 , C_3 and C_4 are the fit coefficients. According to Oliver and Pharr [21], contact depth depends on:

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S} \tag{2}$$

where h_{max} is the maximum indentation depth, P_{max} is the corresponding maximum load, *S* is the contact stiffness determined by the slope of the load-displacement curve at the initial stage of the unloading curve, and $\varepsilon = 0.75$ is the constant of the Berkovich indenter. The hardness of a material is defined as the average pressure exerted by the indenter under the maximum load and is calculated by

$$H = \frac{P_{max}}{A_C} \tag{3}$$

Reduced elastic modulus E_r can be extracted by:

$$E_r = \frac{(\pi)^{1/2}}{2\beta} \frac{S}{(A_c)^{1/2}}$$
(4)

where $\beta = 1.034$ is the shape constant of the Berkovich tip. Since the reduced elastic modulus is the combined response of the indenter tip and the sample, the elastic modulus of the sample *E* can be easily obtained from the following relationship.

$$\frac{1}{E_r} = \frac{1 - v^2}{E} + \frac{1 - v_i^2}{E_i}$$
(5)

where *v* is the Poisson's ratio of the test material, and E_i and v_i are the elastic modulus and Poisson's ratio of the indenter, respectively. For diamond indenters, Frequently used elastic constants $E_i = 1141$ GPa and $v_i = 0.07$ [21,22].

From indentation experiments, the loading-unloading (P-h) curves were recorded. For different parts (HEA particles and ID Layer) in MMC several topic curves were depicted in Fig. 8. As can be seen from Fig. 8, when the indentation depth is shallow (<30 nm) and the *P*-*h* curve shows obvious fluctuation due to the effect of surface oxidation and work-hardening of the sample. After unloading, the curve did not return to the original position, indicating that plastic deformation occurred in the process of loading, resulting in a certain residual pressure. There are no discontinuities in the load-displacement curve in Fig.8, which indicates that there are no cracks or brittle fracture in the sample during the loading process. Obviously, the curves for particles and matrix have much difference, especially, the indentation penetration depth. For HEA particle, the indenter displacement is low and curves are serrated. For ID layer, the indenter displacements are much deeper and increases with the increasing distance from HEA particle.

As can be seen from Fig. 8, creep occurs at the load-holding stage, where the load value remains constant and the indentation depth value increases continuously [23,24]. Compared with the traditional uniaxial creep test, the stress distribution on the surface of the sample under nanoindentation is more complex. Even under the minimum indentation depth, it is dominated by local shear deformation, and the maximum shear stress is usually much higher than the initial yield stress of the material. The non-uniform stress/ strain field under nano-indentation is contrary to the roughly uniform stress/strain field in uniaxial creep test. The non-uniform strain field will lead to the strain gradient [25]. In addition, in the uniaxial tension/compression creep test, the materials involved are limited to the standard distance of the sample [24], and the decrease of strain gradient will lead to the change of indentation depth in the holding stage [26]. This may be the internal reason why the creep of materials in uniaxial creep test usually occurs when the test temperature is close to the melting point of the material, while most high melting materials can creep in the nanoindentation test at room temperature [27-29].

The curves of creep depth and time at different distances from the initial indentation point are given in Fig. 9a, and the surface topography at different indentation positions is shown in the interior plot. From Fig. 9b, it can observe that the creep depth increases with the distance from the high entropy alloy particles. The specific value of creep depth is shown in Fig. 9b. Combining with Fig. 5, it can be seen that the creep depth is related to the distribution of elements in the material. The change of element content and grain size during cooling will change the inherent length of the material, and the strain gradient plasticity theory containing the inherent length of the material will lead to the change of creep depth [30,31]. When the indentation point is located in the interior of the particle, the creep depth does not change greatly, but at the



Fig. 9. (a) Creep depth vs. time at different distances from the initial indentation point; (b) Creep depth and hardness results.

edge of the particle, the creep depth increases from 1.3 nm to 2.8 nm with the increase of Al element. With the decrease of high entropy alloy component elements and the increase of Al elements, the creep depth changes more acutely at the interface, from 2.8 nm to 7.0 nm. On the contrary, the hardness value decreased from 7.9 GPa to 0.9 GPa with the increase of distance. The hardness can be obtained from Eq. (1) and Eq. (3). The value of *c* in Eq. (1) is constant. As the depth of the indentation increases, the load does not change during the load-bearing phase, and A_C increases and the hardness decreases. According to the Tabor's relationship, the indentation hardness *H* is related to the flow stress σ by the constraint factor λ .

$$H = \lambda \sigma$$
 (6)

where the ratio coefficient λ is a constant. Therefore, it is considered that the reduction in nanohardness during the load-holding phase may be due to a decrease in flow stress.

According to the O&P method, elastic modulus *E* and hardness *H* can be obtained from the *P-h* curve of the nanoindentation. In Fig. 10, it is shown the hardness and Young's modulus obtained by the indentation test. In Fig. 10a, it can be seen that the hardness of high entropy alloy is obviously higher than that of the interface layer, and the average hardness of the high entropy alloy particles is 7.5 GPa. The hardness decreases greatly at the interface and the indentation becomes larger and larger, which is due to the fact that the matrix of the diffusion layer is Al and high entropy alloy elements dissolved in Al matrix. Although the hardness of the

diffusion layer is higher than that of aluminum matrix, but still much lower than that of HEA particles. As the indentation position moves to the matrix, the hardness decreases gradually, close to the hardness of the matrix. It is due to that the solid solution elements become less and less with the increase of distance from HEA particles. Young's modulus and hardness have the same trend.

At the junction of the ID layer and the matrix, the hardness and modulus are lower than the hardness and Young's modulus of the matrix. Fig. 11 shows the P-h curves, hardness and modulus of the matrix at different loads. It can be seen from Fig. 11 that under different loads, the size effect of nano-indentation gradually fails and the hardness of the material gradually approach to the true value [32]. It can be seen that the hardness and Young's modulus both decrease with increasing loads. It is inferred that the decrease of hardness and Young's modulus may be caused by two factors: (1) In the process of nano-indentation experiment, the deformation damage occurs due to the local shear stress near the indenter under large load, and the cumulative damage increases the micro-defects and weakens the elastic properties. This in turn leads to the decrease of the Young's modulus of the metal material [33]. (2) The cavities caused by volume shrinkage may be another factor. Volume shrinkage occurs not only during crystallization, but also during cooling after crystallization. After final solidification, some shrinkage cavities may be left behind [31,34].

Comparison has been made for the properties of composites. The hardness of the aluminum matrix, HEAs reinforcements and the composite was measured using a Vickers hardness tester with load of 50gf, and Table 2 shows the results.



Fig. 10. (a) Hardness and (b) Young's modulus of the indentation tests.



Fig. 11. (a) The P-h curves of the matrix; (b) Hardness and (c) Young's modulus at different loading loads.

Table 2	
Hardness of aluminum matrix,	reinforcements and the composite.

	Aluminum matrix	Reinforcements	Composite
Hardness/HV _{0.05}	82.10 78.26 80.61	277.55 280.61 275.51	131.20 127.78 135.48
	80.61	275.51	135.48

The hardness of the composite material is increased nearly 63.7% compared to the hardness of aluminum matrix. The average hardness of the composite is much larger than the $94HV_{0.05}$, which is estimated from the rule of mixture. There are two reasons for this. First, particle reinforcement not only increases the hardness of the matrix, but also the presence of the ID layer, which reduces sintering defects, further increases the hardness of the composite. Second, at the microscopic interface, the hardness shows a gradual change trend. As shown in Fig. 10a, there is a slow transition zone from a low hardnes value to high hardness. In combination with Fig. 5, it can be seen that during the SPS process, the atoms diffuse uniformly in the diffusion layer. A gradient interface microstructure was formed, which is beneficial to reduce the stress concentration at the interface, and improve the stress distribution state and the bearing capacity of the composite. At the same time, the interface and generated compounds hinder the movement of dislocations, causing the dislocations to become plugged, resulting in an increase in the deformation resistance of the material, which is manifested by an increase in the hardness of the composite.

4. Conclusion

 High-entropy alloy particles reinforced aluminum matrix composites were prepared by spark plasma sintering. The composite consists of a matrix, an interdiffusion (ID) layer and HEA particles. The thickness of the ID layer is about $6 \mu m$.

- (2) There is a new generation phase in the ID layer. It has been suggested that the ID layer be affected by the enhanced diffusion reaction under the localized high temperatures. The diffusion layer is divided into inner and outer layers with different elements distribution, which is related to the diffusion coefficient and diffusion mode of elements.
- (3) The ID layer appears obvious creep phenomenon in the indentation process, which is related to the decrease of the bonding force between atoms in the diffusion process. The increase in hardness of the composite (131 HV) is attributed to the enhancement of the particles and the presence of the ID layer.

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Appendix A. Supplementary data

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