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Effect of heat treatment on the interface of high-entropy alloy particles reinforced aluminum matrix composites

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ABSTRACT

In this paper, high entropy alloy particles reinforced 5052 aluminum matrix composites were successfully prepared by vacuum hot pressing sintering technology. Interface layers with different thickness were formed in the composites by heat treatment. The effects of heat treatment temperature and time on the interface layer of composites were systematically studied by scanning electron microscopy (SEM), energy dispersion spectroscopy (EDS), X-ray diffraction (XRD), electron probe microanalysis (EPMA) and nano-indentation. The results show that the new core-shell structure particles are dominant in the composites with long holding time at 500 °C heat treatment. Hardness and Young's modulus of the composites are obviously improved by the generation of the interface layer. With the increase of heat treatment time, the thickness of the interface layer increases, and the discontinuous interface layer will obviously increase the hardness and Young's modulus of the composites, which is related to the stress concentration and the bonding degree between the interface and the matrix.

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1. Introduction

Particle reinforced aluminum matrix composites have the advantages of high specific strength and specific stiffness, good thermal stability, good thermal conductivity, wear resistance and corrosion resistance [1]. At present, the common particles reinforcement of aluminum matrix composites are ceramic particles and graphite particles or amorphous particles and so on [2–5], but the wettability of these reinforced particles with aluminum matrix does not reach the ideal state. When the ceramic phases are used as the strengthening phase, the diffusion of the strengthening phases are difficult, so that it is not completely miscible with the metal matrix, and the interface bonding is poor. When the amorphous phases are used as the strengthening phases, the amorphous phases are not a stable phase, and the amorphous phases are prone to

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crystallization at high temperature, which destroys the interfacial stability of the composites and makes it prone to fracture [6].

In recent years, high-entropy alloys (HEAs) have attracted attention due to their unique multi-component solid solution structure and their excellent comprehensive properties [7]. Due to the natural interfacial bonding between particles and matrix, the interfacial wettability and compatibility of high entropy alloy particles reinforced metal matrix composites are better than that of ceramic particles. At present, high entropy alloy as a reinforcement of composites has achieved some results. Meng et al. used the laser melting technology (LMI) to prepare AlCoCrCuFeNi particles reinforced AZ91D magnesium matrix composites, and found that the friction and wear properties of the composites were significantly improved [8,9]. Praveen et al. added Al-20Cu-10Mg high-entropy alloy to the 2024 aluminum alloy matrix by stirring casting, which greatly improved the mechanical properties such as yield strength, tensile strength and Young's modulus [10]. Tan et al. prepared Al_{0.6}CoCrFeNi particles reinforced Al-based amorphous composites by spark plasma sintering, and the yield strength measured by compression test was as high as 3120 MPa [11]. Liu et al. prepared





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AlCoCrFeNi particle reinforced aluminum matrix composites by spark plasma sintering. They found that the thickness of the interface layer increased with the increase of sintering temperature, and the yield strength and plasticity of the composites were significantly improved [12]. Chen et al. prepared AlCoNiCrFe particles reinforced copper matrix composites by vacuum hot pressing [13]. It was reported that there was no obvious interfacial reaction between copper matrix and high entropy alloy reinforced particles. The yield strength of the material was similar to the Voigt upper limit calculated by the model.

In the previous work [14], there were obvious interdiffusion layers in CoCrFeMnNi particle reinforced aluminum matrix composites prepared by spark plasma sintering. At present, there are few researches on the preparation of high-entropy alloy particles reinforced aluminum matrix composites by vacuum hot pressing. In this paper, the 5052Al which cannot be strengthened by heat treatment is used as the matrix, and the Al_{0.6}CoCrFeNi particles are used as the reinforcing material to prepare the aluminum matrix composite. The interface properties of the composites were studied by heat treatment process, and the effects of the interfaces on the mechanical properties of the composites were investigated.

2. Experimental

2.1. Samples preparation

Using commercially available 5052Al alloy powder (<50 µm, composition shown in Table 1) and Al_{0.6}CoCrFeNi powder(38–75 μ m) as starting materials, the mixed powder is uniformly mixed in a vacuum atmosphere with a ball-to-powder weight ratio of 10:1 and a rotating speed of 200 rpm in a planetary ball mill. The volume fraction of the HEA powder was determined to be 7%, and the composite powder was sintered in a vacuum hot pressing furnace at 823 K and 30 MPa for 60 min. The billet of 20 $\text{mm} \times 10$ mm was obtained after sintering, and the billet was heat-treated in a resistance furnace. Respectively, the heat treatment scheme was air-cooled after being kept at 350 °C, 500 °C and 600 °C for 24 h. In addition, in order to study the effect of heat treatment time on composite materials, the holding time was added for 12 h and 48 h at 500 °C. The phase composition of the composites was measured by X-ray diffractometer (XRD). The microstructure was characterized by metallographic microscope and scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS). The diffusion layer was quantitatively analyzed by electron probe. The alloy was mechanically polished to mirror finish prior to sample testing.

2.2. Nanoindentation experiment

Nanoindentation was performed on the sintered composite at room temperature using a Hysitron-Picoindenter and berkovich triangular diamond tip. Arbitrarily select the reinforcement particles, indentation test from the matrix to the particle direction, set the indentation load to 3000 μ N and 4000 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 1.5 μ m.

Table 1
Chemical composition of 5052 aluminum alloy.

Element	Mg	Fe	Cr	Si	Mn	Zn	Cu	Al
wt.%	2.2-2.8	0.4	0.15-0.35	0.25	0.1	0.1	0.1	Bal.

3. Results and discussions

3.1. Microstructure

Fig. 1 shows metallographic pictures at different heat treatment temperatures and times. Dark gray Al_{0.6}CoCrFeNi particles and black second phase particles are randomly distributed in the aluminum alloy matrix, and the high entropy alloy particles are spherical and do not deform during ball milling and sintering. It can be seen from Fig. 1a that there is a clear boundary between the particles and the matrix, which indicates that there is no obvious interfacial reaction between HEA and the substrate during sintering. As the heat treatment temperature and time change, the interface between the particles and the substrate also changes significantly. In Fig. 1 b, there is no obvious interfacial reaction between the particles and the matrix, and the second phase particles and grain size have not changed significantly. This is because the temperature is too low and the atomic diffusion activation energy is not reached, so that the 5052Al alloy is not homogenization annealing occurs. It can be seen from Fig. 1 c-e that at the heat treatment at 500 °C, the thickness of the interface layer increases significantly with the increase of the holding time, but there is no significant difference in the interface thickness between 24 h and 48 h, which is related to the solid solubility. At the same time, the second phase also increases obviously, because the increase of temperature will greatly accelerate the diffusion process and increase the degree of homogenization. When the heat treatment is performed at 600 °C, the aluminum alloy undergoes a significant overburn phenomenon due to the dissolution of the second phase and the formation of a liquid phase.

Fig. 2 shows the XRD patterns of composites at different heat treatment times and temperatures. As can be seen from Fig. 2a, the Al_{0.6}CoCrFeNi high-entropy alloy only has a single-phase BCC solid solution structure (within this scan angle range), which is related to the particle size [15]. In addition to the matrix Al phase and a small amount of BCC phase, a new Al(Co, Fe) phase and an Al₁₂Mg₁₇ phase are formed in the sintered product of the composite. It can be seen that no new phase is formed during heat treatment, and the diffraction peak strength of Al(Co, Fe) phase and Al₁₂Mg₁₇ phase in the composites increases slightly. As the heat treatment time and temperature increases, the position of diffraction peak also appears different amplitude shift, which may be due to the lattice distortion caused by the diffusion of high entropy particle elements into Al alloy during heat treatment.

3.2. Interface layer analysis

Fig. 3 shows the element mapping results of the original composites, there is a clear boundary between the reinforcement particles and the matrix. The difference of element concentration does not cause interdiffusion between the high-entropy alloy particles and the matrix, which is consistent with the result of Fig. 1a. This is attributed to two factors. Firstly, due to the particularity of the crystal structure of high entropy alloys, the serious crystal lattice distortion hinders the diffusion of atoms and slows the diffusion of high entropy alloy elements to the matrix. Secondly, due to the relatively large radius of Al atoms, during the sintering process, it cannot to provide enough energy for Al atomic radius to overcome the potential energy barrier to replace other atoms in the crystal structure of high entropy alloy. It can be seen from the figure that the high entropy alloy elements are dispersed and distributed in the matrix and combine with the matrix elements to form a new phase in the sintering process. Here is the second phase particles in the matrix, because the element content here is lower than the reinforcement, the color is dim. Which is consistent with the XRD



Fig. 1. Metallographic images of composites under different heat treatment conditions. (a) Original sample; (b) 350 °C + 24 h; (c) 500 °C + 12 h; (d) 500 °C + 24 h; (e) 500 °C + 24 h; (f) 600 °C + 24 h.



Fig. 2. XRD patterns of Composites under different Heat treatment conditions. (a) Heat treatment at different temperatures at 24 h; (b) Heat treatment at 500 °C at different time.



Fig. 3. Elemental distribution of the initial composite.

results. The distribution of different elements in the matrix is quite different, and the reasons are described in the following contents.

In order to study the interface layer, composite materials with different interface characteristics at different heat treatment times at 500 °C were selected, and the composition of the interface layer was tested in detail. Fig. 4 shows the elements distribution image of the composite under different heat treatment times. There are some differences in the distribution of aluminum, iron, chromium, cobalt and nickel in the diffusion layer. This is due to the hysteresis effect in the diffusion kinetics of high entropy alloy and the different diffusion rate of each element in the high entropy alloy, which leads to the difference of the content and distribution of each element in the interface layer. Compared with other elements, the distribution and content of Cr in the composites are lower, which may be due to the larger diffusion coefficient of iron and nickel in aluminum, which leads to the diffusion of iron and nickel into the matrix. The diffusion of Fe and Ni element will inevitably occupy part of the diffusion channel nearby, which will cause the relative diffusion channel of Cr element to be relatively reduced, thereby slowing the outward diffusion rate of Cr element and even hindering the outward diffusion of Cr element. Four kinds of contrast are reflected in Fig. 4a. The gray is the 5052 Al matrix, the white is the Al_{0.6}CoCrFeNi high entropy particles, the bright gray is the interface layer between the matrix and the reinforcement after heat treatment, and the black is the second phase precipitated during heat treatment. Compared with Fig. 4b, due to the short heat treatment time, the interface layer in Fig. 4a is thinner, and the element distribution of the interface layer is not obvious. Combined with the XRD results, it can be determined that the formation of the interface layer is attributed to the solid/solid diffusion between the HEA particles and the matrix.

In order to analyze the specific components of the interface layer, the chemical composition of the spots marked in Fig. 4 was analyzed by an electron probe, and the results are shown in Table 2. In order to better observe the law of element distribution, Fig. 5 is made according to Table 2. It can be seen from Fig. 5 that the elements distributions under different heat treatment times have the same distribution law. Compared to other elements, the contents of cobalt, iron and nickel are significantly higher with distance from the particles. This may be due to the fact that the diffusion coefficients of Fe and Ni are larger than those of other elements in aluminum, and according to the binary phase diagram of iron, nickel and aluminum, The temperature range in which they react with aluminum to form intermetallic compounds is lower than that of other high entropy alloy elements, which indicates that iron and nickel elements diffusion in the form of atoms for a longer time, so the diffusion distance is relatively long. So it is distributed over the entire aluminum substrate.

It can be clearly seen from Fig. 4 that as the heat treatment time increases, the thickness of the diffusion layer gradually increases and exhibits an uneven distribution, and the diffusion layer completely surrounds the reinforcement particles, and at the same time, the shape of the high-entropy alloy particles gradually changes irregular, this is related to the proliferation process. In



Fig. 4. Elemental distribution of composite materials at different heat treatment times at 500 °C (a) 12 h; (b) 24 h

Table 3

aDIC 2	
hemical composition of the composite interface layer (at	. %).

Regions	Al	Fe	Zn	Mg	Mn	Cu	Si	Cr	Ni	Со
Α	77.022	4.937	0.0883	1.3544	1.189	0.0383	3.344	4.915	3.646	3.466
В	80.322	4.451	0.0187	2.053	0.0828	0.1029	0.8826	0.809	6.316	4.962
С	80.467	3.432	0.0244	5.477	0.0463	0.1392	0.3581	3.652	2.585	3.819
D	80.335	3.154	0.0763	5.823	0.0579	0.1177	0.2781	3.784	2.707	3.667
E	80.733	3.869	0.1413	4.493	0.056	0.0564	0.2193	2.846	3.977	3.609
F	81.24	5.717	-	1.0889	0.0356	-	0.241	0.3255	5.715	5.637



Fig. 5. The distribution of the elements shown in Table 2.

order to explore the relationship between the interface thickness and the particles, taking the particle morphology as the boundary, the maximum inner tangent circle of the irregular pattern is obtained [16]. The center of the inner tangent circle is the center point, and 12 directions are selected as the representative, and the blue arrow is taken as the starting point. The distance from the particle boundary to the inner tangent circle and the thickness of the interface layer in this direction are measured clockwise, and the relationship between the interface thickness and the particle boundary is quantified, and the results are shown in Fig. 6. It can be seen from the figure that as the ratio of the interface to the particle thickness decreases, the thickness of the interface layer increases, and it is guessed that this change is related to the curvature of the particle. Although the reinforcement particles are similar to spherical particles, the curvature of the particle boundary is not completely consistent. Because there is no mutual diffusion between the particles and the matrix, the high entropy alloy particles can be regarded as a diffusion source. The matrix around the particles can be regarded as a workpiece with a radius of curvature R. When R is positive, the area perpendicular to the diffusion direction decreases continuously, so that the infiltrated elements tend to gather inward, and the surface infiltration concentration increases and the layer depth increases. When R is negative, the area perpendicular to the diffusion direction increases, so that the infiltrated elements have the trend of inward diffusion, so the surface infiltration concentration decreases and the layer depth decreases. The conclusion is consistent with the appearance shown in Fig. 6.

3.3. Micromechanical properties

The particles with good interface characteristics were selected for nano-indentation test. Fig. 7 shows the indentation diagram at 500 °C for different heat treatment time. The indentation points are arranged tightly at a distance of 1.5 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 matrix to the particles, the indentation morphology becomes smaller and the plastic deformation decreases.

The principle of nano-indentation test was proposed by Olive and Pharr [17]. The hardness and elastic modulus of the sample were obtained by measuring and recording the continuous loaddepth curve.

$$E_r = \frac{(\pi)^{1/2}}{2\beta} \frac{S}{A^{1/2}}, \ \frac{1}{E_r} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$
(1)

Where *S* is the contact stiffness, $s = \frac{dP}{dh} | h = h_{max}$; E_r is the equivalent elastic modulus; E_i , v_i are the elastic modulus and Poisson's ratio of the indenter respectively; E, v_i are the measured elastic modulus and Poisson's ratio; β is the shape parameter of the indenter; *A* is the contact area. For the Berkovich indenter herein, $\beta = 1.034$, $E_i = 1141$ GPa and $v_i = 0.07$ [17,18].

The contact depth is related to the maximum indentation depth and is defined as:

$$h_c = h_{max} - h_d = h_{max} - \varepsilon \frac{P_{max}}{S}, \quad A = 24.56h_c^2 \tag{2}$$

Where: h_d is the elastic recovery depth; ε is the indenter geometry parameter; P_{max} is the maximum indentation load. Hardness is the ratio of the maximum indentation load to the contact area,

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

Firstly, a load of 3000 µN was selected to perform a nanoindentation test on the sample that was heat treatment at 500 °C for 12 h, and the *p*-h curve of different regions of the composite material (particle-interface layer-matrix) were obtained (as shown in Fig. 8). It can be seen from Fig. 8 that there is a displacement bursts phenomenon in the loading curve of high entropy alloy particles [19,20]. That is caused by two reasons. First, there are crystal defects below the surface of the high-entropy alloy particles. During the loading process, the presence of vacancies under the indenter will cause a displacement bursts at the loading curve. Second, for the nanoindentation of the face centered cubic crystals, when the maximum shear stress generated under the indenter is of the order of the theoretical shear stress, this high local stress seems to cause homogeneous nucleation of dislocations under the surface, thereby displacement burst [21]. The indentation curves at the granules show good coincidence and therefore tend to be the second reason. For Al_{0.6}CoCrFeNi high entropy alloy, there are two phases, FCC and BCC phase. For particles size <75 µm, there is no FCC phase at the scanning angle of 20–90°, while the FCC phase exists in other scan angles. There are obvious differences in the loading curves at the interface layer, the indentation depth



Fig. 6. Relationship curves between interface thickness and particles: (a) 12 h; (b) 24 h



Fig. 7. Metallographic diagram of the indentation of the composite material from the Al matrix to the HEA particles at different time during 500 °C heat treatment: (a) 12 h; (b) 24 h.

increases gradually along the direction of diffusion, and the curve fluctuates obviously, which is related to the roughness of the interface, and moves with the direction to the matrix. The fluctuations are getting more and more intense. The loading curves at the substrate are smooth, there is no obvious fluctuation, and the indentation depth near the interface layer is shallow, which is due to the existence of compressive stress at the interface and substrate interface, so that the hardness is improved. According to the above equation, the hardness and Young's modulus of the composite are calculated, as shown in Fig. 9. In Fig. 9a, it can be seen that the hardness of the high-entropy alloy is higher than that of the Al matrix, and the hardness value at the particles does not change drastically. The hardness value is about 10 GPa. When the indentation position moves to the interface, the hardness decreases greatly, and gradually decreases along the direction of the matrix. The hardness of the interface is still higher than that of the matrix, and the hardness fluctuates up and down in 6 GPa. The hardness of the substrate drops suddenly again, and gradually decreases with the movement of the indentation position, and stabilizes around 1.8 GPa. Young's modulus and hardness have the same trend. Young's modulus at the granules is about 280 GPa, and Young's modulus at the interface changes with the element content at the interface, varying between 120 and 145 GPa. Young's modulus at the matrix is about 80 GPa.

According to the study of Xia et al. [22], when the indentation depth is shallow (<100 nm), the Young's modulus will have a large deviation from the standard value, which is related to the first contact error. Eqs. (1)-(3) are integrated into equation (4).



Fig. 8. The *p*-*h* curves of composites heat treated at 500 °C for 12 h.

$$E = \frac{1 - v^2}{(1 - E_i) - \left(\frac{1 - v_i^2}{E_i}\right)} = \frac{1 - v^2}{\left(\frac{2\sqrt{24.56}h_c}{S\sqrt{\pi}}\right) - \left(\frac{1 - v_i^2}{E_i}\right)}$$
(4)

When the indentation depth is deep (>100 nm), the calculated Young's modulus is consistent with the Young's modulus of the material itself. In order to eliminate the contact error, a larger force (4000 mN) was selected to carry out nano-indentation test on the samples heat-treated for 24 h. Fig. 10 shows the *p*-h curves of the composite, and the indentation depth is more than 100 nm. The loading curve has no obvious fluctuation. The hardness H and Young's modulus E of the composite are calculated by using the above equation, and the results are shown in Fig. 11. The Young's modulus of the particles is about 160 GPa, which is much smaller than that of heat treatment for 12 h. Due to the increase of indentation depth, the contact error decreases, and the modulus result is closer to the real value. Young's modulus of interface and matrix are 123 GPa and 78 GPa, respectively. The hardness of the substrate decreased to about 1.6 GPa. The results obtained are consistent with the results of Fig. 9. With the indentation moving towards the matrix, the modulus and hardness decrease gradually, which is caused by the gradient distribution of high entropy alloy elements in the process of heat treatment. Comparing Fig. 9 and 11, it can be found that Young's modulus of the interface layer under different heat treatment time has consistency, fluctuating up and



Fig. 10. The *p*-*h* curves of composites heat treated at 500 °C for 24 h.

down at 120–130 GPa, and the hardness is also consistent, fluctuating up and down at 5–6 GPa. Since the roughness does not have a large influence on the macrohardness and Young's modulus of the material, the heat treatment time only affects the thickness of the interface layer, and has no significant effect on the performance of the interface layer. The matrix properties of the composites are higher than the theoretical values calculated by the mixing law of the composites, which is due to the fact that the interface hinders the movement of dislocations and leads to the accumulation of dislocations. It increases the deformation resistance of the composites and improves the hardness and Young's modulus of the composites.

To investigate the effect of heat treatment time on the properties of the composite, Fig. 12 shows the *p*-*h* curve of the original sample and Fig. 13 shows the hardness and Young's modulus of the calculated composite. It can be seen from the diagram that Young's modulus of the particles is about 190 GPa, and the modulus of the matrix is 77 GPa, which is close to the real value. The hardness of the particles does not change much from that after heat treatment, about 10 GPa. The hardness of the matrix is obviously lower than that of the sample after heat treatment, which is about 1.43 GPa. In order to compare the properties of the composites, Table 3 shows the modulus and macro and micro hardness of the composites under different heat treatment conditions. It can be seen from the



Fig. 9. (a) Hardness and (b) Young's modulus of the indentation tests for composites heat-treated at 500 °C for 12 h



Fig. 11. (a) Hardness and (b) Young's modulus of the indentation tests for composites heat-treated at 500 °C for 24 h



Fig. 12. The *p*-*h* curves of Original sample.

table that the existence of interface has little effect on Young's modulus and great influence on hardness, and the hardness increases by 14.7%–26.6% under different heat treatment conditions.

In this work, the performance of the interface layer remains basically the same, but as the thickness of the interface gradually increases, the hardness and Young's modulus of the composite material decrease. The main reason for this is that during the heat treatment, the formation of a discontinuous interfacial layer causes a large concentration of stress in the matrix, which increases the hardness and Young's modulus of the matrix. As can be seen from Fig. 4, when the heat treatment time increases, the interface layer gradually becomes continuous and well bonded to the matrix, forming a distinct core structure [23], macroscopic cracks are greatly reduced, and stress is released. At the same time, the interface area between the matrix and the particles is increased, and more load can be conducted from the substrate to the particles, so that the hardness and Young's modulus of the composite are lowered.



Fig. 13. (a) Hardness and (b) Young's modulus of the indentation tests of original sample.

Table 3

Young's modulus and macro and micro hardness of the composites under different heat treatment conditions.

	Original sample	heat treated at 500 $^\circ\text{C}$ for 12 h	heat treated at 500 $^\circ\text{C}$ for 24 h
Young's modulus/GPa	77	80	79
Hardness/GPa	1.43	1.81	1.60
Hardness/HV _{0.05}	136	178	155

4. Conclusions

In this paper, high entropy alloy particles reinforced aluminum matrix composites were successfully prepared by vacuum hot pressing technology. The following conclusions are drawn:

- (1) The composites sintered at 550 °C are composed of matrix and HEA particles, and the interface layer of core-shell structure is formed after heat treatment at temperatures above 500 °C. The hardness and Young's modulus of the composite are improved by the formation of interface layer.
- (2) There is no new phase in the interface layer, and the thickness of the interface layer is related to the heat treatment temperature and time, which is related to the diffusion coefficient and diffusion mode of the element. Compared with the complete interface layer, the discontinuous interface layer will obviously increase the hardness and Young's modulus of the composites, which is related to the stress concentration and the bonding degree between the interface and the matrix.

Declaration of competing interest

The authors declared that they have no conflicts of interest to this work.

We declare that we do not have any commercial or associative interest that represents a conflict of interest in connection with the work submitted.

CRediT authorship contribution statement

Zhanwei Yuan: Resources, Writing - review & editing, Supervision, Data curation. **Wenbin Tian:** Software, Investigation, Writing - original draft. **Fuguo Li:** Methodology, Writing - review & editing. **Qinqin Fu:** Investigation. **Xingang Wang:** Methodology, Writing - review & editing. **Weifeng Qian:** Investigation. **Wangcong An:** Investigation.

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