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ARTICLE

Effect of Boron on the Microstructure, Phase Assemblage and Wear Properties of $Al_{0.5}CoCrCuFeNi$ High-Entropy Alloy

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Abstract: The microstructure, phase assemblages, hardness and wear properties of $Al_{0.5}CoCrCuFeNiB_x(x=0\sim1)$ high-entropy alloys were investigated. The results show that without boron addition, the alloy is composed of simple fcc solid solution structure. In the alloys with boron additions, boron precipitates as borides instead of entering the crystal structure and thus there is no change in the lattice constant of the matrix fcc phase. Borides precipitation significantly hardens the alloys and the hardness shows strong correlation with the boron content. There is no distinct difference in the wear resistance of the alloys with $x\leq0.4$, but the wear resistance increases linearly with the boron content increasing in the alloys with $x\geq0.6$. With the boron content increasing, the wear mechanism changes from delamination wear to oxidative wear. The great enhancement in the hardness and wear resistance is attributed to the combination of the large hard borides and ductile and tough fcc matrix.

Key words: high-entropy alloys; borides; hardness; wear resistance

Traditional alloys are based on one principle element taking up more than 50 at% with the alloying elements making use of the edge components region in the phase diagrams for performance and/or processing enhancement [1,2]. In traditional alloys, the amount of alloying elements should be minor to avoid the formation of complicated intermetallic compounds which result in brittleness, difficulty in processing and challenging analysis. High-entropy alloys (HEAs) with multiple principal elements in equiatomic or close-to-equiatomic ratios are a novel alloy system developed beyond the traditional metallurgical design [3,4]. In such a highly concentrated multi-component alloys system, simple solid solutions with fcc and/or bcc structures tend to form instead of intermetallic compounds and elaborate phases during solidification[5-7].

Since the development of HEAs, extensive trials have led to many alloys with simple crystal structures and exceptional properties [8-11]. The $Al_{0.5}CoCrCuFeNi$ alloy consists of simple fcc solid-solution structure and is ductile, work hardenable, and strong even at temperature up to 800 °C [12-14]. In the present study, to further tune the properties, varying contents of boron were added to $Al_{0.5}CoCrCuFeNi$ alloy and the wear resistance was evaluated under drying sliding wear test. The effects of boron addition on the microstructures, phase assemblages and their formation mechanism of the alloys were discussed.

1 Experiment

Elemental Al, Co, Cr, Cu, Fe and Ni granules with purity higher than 99.9% and Fe-28.9B (wt%) master alloy were used as raw materials. Alloy ingots with nominal composition of $Al_{0.5}CoCrCuFeNiB_x(x=0\sim1.0)$ were prepared by vacuum arc melt casting in high-purity argon atmosphere with a water-cooled copper crucible. The ingots were re-melted 5 times to improve the chemical homogeneity.

The crystal structure of the samples was characterized by X-ray diffractometer (XRD) for a 2θ range of 20° to 100° with a scanning rate of 2°/min. Scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) were used

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to analyze the microstructures and chemical compositions. Vickers hardness was measured with a load of 50 g and a duration time of 15 s for each measurement. The adhesive wear behavior of the alloys was investigated by pin-on-disk sliding with a normal load of 100 N under dry sliding conditions at room temperature. The wear test specimens were pins 6 mm in diameter and 12 mm in height. During the test, each pin was fixed in a holder to wear against a rotating $C_{12}MoV$ steel disk which has a diameter of 70 mm and a hardness HV value of 7600 MPa. The distance between the pin and the center of the disk was 30 mm and the sliding speed was 0.8 mm/s.

2 Results

2.1 Microstructure and phase assemblages

The crystal structures of the as-cast $Al_{0.5}CoCrCuFeNiB_x$ alloys are shown in Fig.1. Only XRD diffraction peaks corresponding to a fcc crystal structure are identified in the alloy without boron addition. After the addition of boron, the matrix phases of the alloys are mainly composed of fcc structure, but peaks corresponding to borides are observed. For the alloys with $x \geq 0.8$, peaks corresponding to an ordered fcc phase appear. For the alloy without boron addition, the lattice constant is 0.3595 nm. For the alloys with the additions of boron, the lattice constants are estimated to be 0.3592 ± 0.0007 nm.

Fig.2 shows the back-scattered images of the as-cast $Al_{0.5}CoCrCuFeNiB_x$ alloys. Table 1 shows the EPMA results on the chemical compositions of phases as indicated in Fig.2. The alloy without boron addition has a typical cast dendrite (A) and interdendrite (B). Both the dendrites (A) and interdendrites (B) are basically composed of multiprincipal elements and the interdendrite regions are copper-riched. With the addition of boron, stringer-shaped precipitates (C) begin to precipitate from both the dendrites and the interdendrites. The stringer-shaped precipitates are (Cr, Fe, Co)B-type boride which is poor in Al, Cu and Ni. The enrichment of elements in this boride follows Cr>Fe>Co. With increasing boron content, the borides become larger and the copper-rich interdendrites gradually disappear.

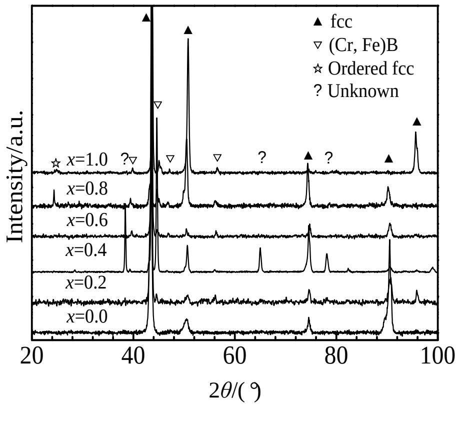

Fig.1 XRD patterns of as-cast $Al_{0.5}CoCrCuFeNiB_x$ ($x = 0$, 0.2, 0.4, 0.6, 0.8, 1.0) alloys

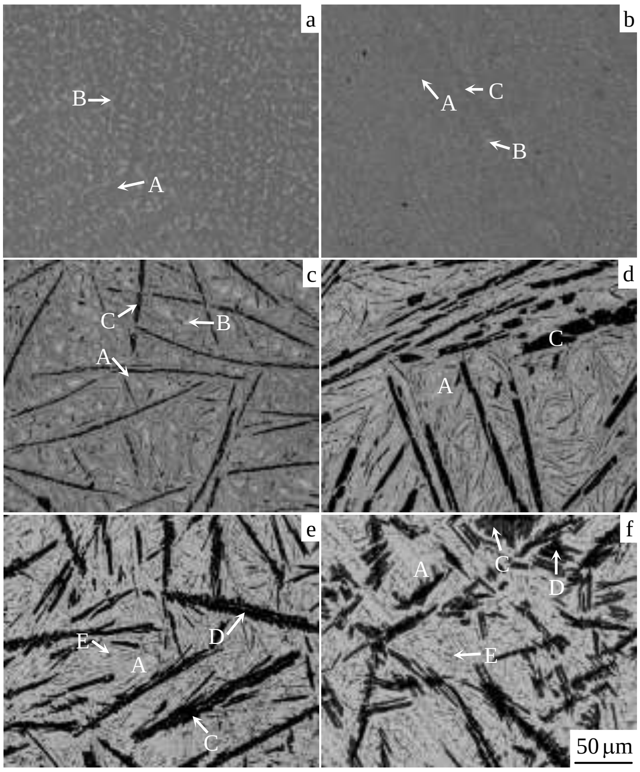

Fig.2 SEM images of as-cast $Al_{0.5}CoCrCuFeNiB_x$ alloys: (a) $x = 0$, (b) $x = 0.2$, (c) $x=0.4$, (d) $x = 0.6$, (e) $x = 0.8$, and (f) $x = 1.0$

In the alloy with higher boron contents ($x \geq 0.8$), there are other two phases observed besides the stringer-shaped (Cr, Fe, Co) B-type borides. The one with black color is CrB-type borides (D) which are located at the center of (Cr, Fe, Co) B-typed borides and the other one with Chinese script-shape is (Co, Fe, Cr, Ni)B-type borides (E). The enrichment of elements in (Co, Fe, Cr, Ni) B-typed borides follows Co> Fe>Cr>Ni.

2.2 Hardness and wear resistance

Fig.3 shows the hardness and wear resistance of the $Al_{0.5}CoCrCuFeNiB_x$ alloys as functions of boron content. The hardness values of the alloys exhibit strong correlation with the boron content and increase linearly as x is varied from 0 to 1.0. The hardness HV of the alloy with $x=1.0$, 6070 MPa, is 2.31 times higher than that of the alloy without the addition of boron, 2630 MPa. There are no distinct differences in the wear resistance for the alloys with $x \leq 0.4$; however, the wear resistance is significantly enhanced and exhibits strong correlation with the boron contents when $x \geq 0.6$. The wear resistance of the alloy with $x = 1.0$, 2.05 km/mm^3, is about 20 times higher than that of the alloy with $x=0$, 0.10 km/mm^3.

Fig.4 presents the wear resistance as a function of hardness of the $Al_{0.5}CoCrCuFeNiB_x$ alloys. The wear resistance versus hardness of the conventional H13 steel is also plotted for

Table 1 EPMA analysis on chemical composition of zone indicated in Fig.2 for as-cast $Al_{0.5}CoCrCuFeNiB_x$ alloys (at%)

x	Zone	Al	Co	Cr	Cu	Fe	Ni	B
0	Nominal	9.09	18.18	18.18	18.18	18.18	18.18	-
	A	7.38	23.73	21.31	9.73	20.83	17.02	-
	B	13.37	3.64	2.56	69.80	3.30	7.33	-
0.2	Nominal	8.77	17.54	17.54	17.54	17.54	17.54	3.51
	A	11.21	19.44	14.59	16.20	19.22	19.34	0.00
	B	7.59	15.78	18.99	14.39	16.50	11.97	14.79
0.4	Nominal	8.47	16.95	16.95	16.95	16.95	16.95	6.78
	A	7.89	22.55	14.32	13.84	22.95	18.35	0.00
	B	0.04	4.69	50.47	0.27	9.01	0.04	35.47
0.6	Nominal	8.20	16.39	16.39	16.39	16.39	16.39	9.84
	A	9.50	22.87	9.26	17.17	20.90	20.29	0.00
	B	0.04	6.34	51.04	0.24	11.65	0.13	30.59
0.8	Nominal	7.94	15.87	15.87	15.87	15.87	15.87	12.70
	A	11.83	21.40	5.34	20.43	20.06	20.94	0.00
	C	0.03	3.13	53.64	0.21	6.21	0.08	36.70
	D	0.02	0.57	52.99	0.12	1.91	0.00	44.38
	E	0.19	33.20	6.21	0.57	26.39	5.87	27.58
1.0	Nominal	7.69	15.38	15.38	15.38	15.38	15.38	15.38
	A	12.47	19.12	3.19	26.38	16.93	21.91	0.00
	C	0.02	11.02	42.85	0.47	18.03	0.12	27.49
	D	0.02	0.87	55.14	0.15	2.53	0.01	41.27
	E	4.16	29.39	11.12	2.04	29.92	5.61	17.75

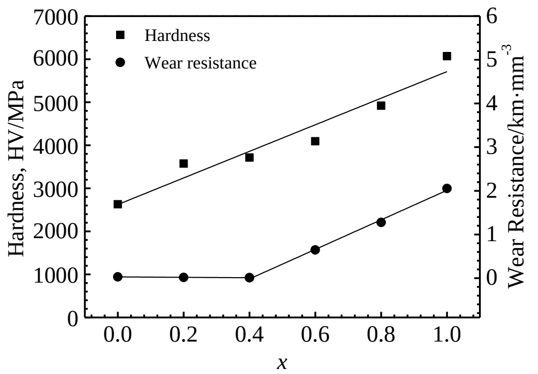

Fig.3 Hardness HV and wear resistance as functions of boron content of $Al_{0.5}CoCrCuFeNiB_x$ alloys

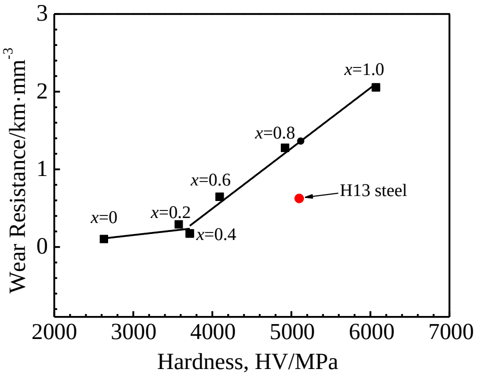

Fig.4 Wear resistance as a function of hardness of $Al_{0.5}CoCrCuFeNiB_x$ alloys

comparison. The variations in wear resistance show the almost same tendency as in Fig.3. When $x\leq0.4$, both the hardness and wear resistance of the alloys are lower than those of H13 steel. When $x=0.6$, the alloy has lower hardness but similar wear resistance compared to H13 steel. When $x\geq0.8$, the alloys show much higher wear resistance than H13 steel even though the hardness of the alloy with $x=0.8$ is comparable to that of H13 steel.

2.3 Wear mechanism

Fig.5 shows the morphologies of the worn surface of the alloys after the test and the debris produced during the test. For the alloy without boron addition, the worn surface is grooved and noticeable plastic deformation features along the grooves are seen. The debris of the alloy without boron addition has a flake-like shape and is 300~1000 μm in size. The flake-like shape indicates that the debris is worn off from $Al_{0.5}CoCrCuFeNi$ and the adhesive wear mechanism of $Al_{0.5}CoCrCuFeNi$ is predominantly of delamination wear [12]. The chemical composition of both the worn surface and wear debris in Table 2 shows that they all contain a significant

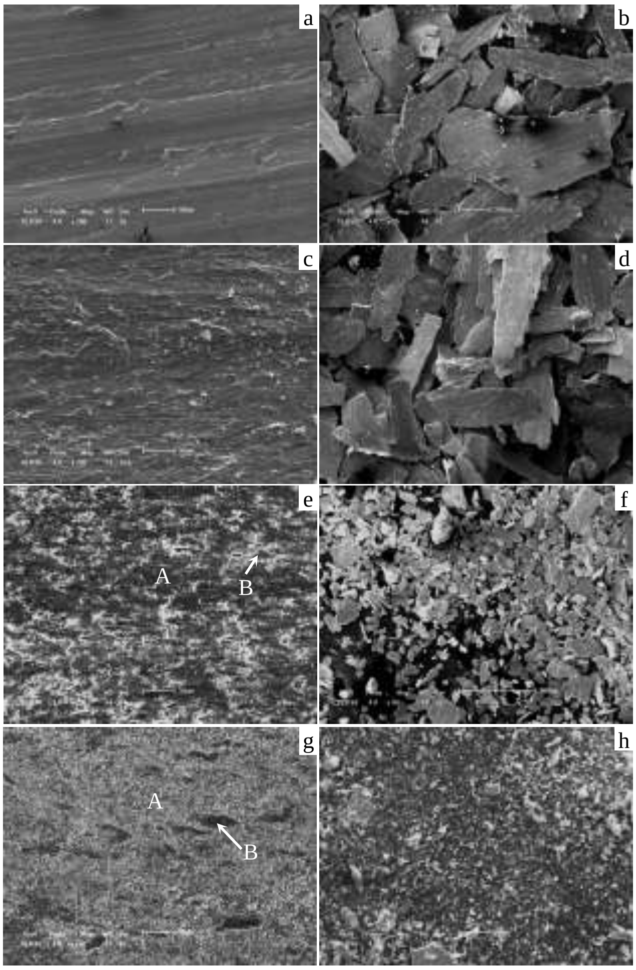

Fig.5 SEM morphologies of wear surface of $Al_{0.5}CoCrCuFeNiB_x$ alloys: (a, b) $x=0$, (c, d) $x=0.4$, (e, f) $x=0.6$, and (g, h) $x=1.0$

amount of oxygen indicating oxidation occurs on the worn surface heated by friction and deformation.

For the alloys with lower boron content ($x \leq 0.4$), plastic deformation and plowing grooves on the worn surface can be seen. Materials loss in the alloys with $x \leq 0.4$ seems to have occurred primarily by microploughing due to the plastic deformation induced by the hard asperities of the $Cr_{12}MoV$ counterbody and work-hardened wear debris. The plastic features indicate that the matrix fcc phase is still ductile and the finer grooves on the worn face reveal the occurrence of abrasive abrasion. The debris is smaller than that of the alloy without boron addition, around 200~800 μm in size, and also has a flake-like shape. The oxygen contents of the worn surface and debris are all higher than those of the alloy without boron. It is indicated that the wear type of the alloy with lower boron content is predominantly of abrasion and oxidative wear. An increase in boron content to $x \geq 0.6$ results in a smoother worn surface, especially the alloy with $x=1.0$. The plastic deformation and groves are hardly seen on the worn surface, justifying the higher wear resistance observed here before. The boride particles on the worn surface are clearly seen and material is transferred from the counterpart to the specimen during the wear test. Wear debris has a particle-like shape and is much finer, around 100 μm in size for the alloy with $x=0.6$, 10 μm in size for the alloy with $x=1.0$. The oxygen concentration of wear debris is much more than those of the alloys with $x \leq 0.4$. All these indicate that the wear mechanism is predominantly of oxidative wear.

3 Discussions

Currently, the formation of simple microstructure, fcc and /or bcc solid solutions, instead of complicated intermetallic

Table 2 Chemical composition of worn surface and wear debris of the $Al_{0.5}CoCrCuFeNiB_x$ alloys by EPMA analysis in Fig.5 (at%)

x	Zone	Al	Co	Cr	Cu	Fe	Ni	B	O
0	Nominal	9.09	18.18	18.18	18.18	18.18	18.18	-	-
	Worn surface	9.74	16.80	15.22	13.98	17.24	14.87	-	12.16
	Debris	8.12	15.67	14.14	13.59	13.39	14.16	-	20.94
0.4	Nominal	8.47	16. 59	16.95	16.95	16.95	16.95	6.78	-
	Worn surface	6.86	14.42	21.16	12.95	12.72	10.50	0.00	21.39
	Debris	9.31	15.08	9.33	13.48	13.67	15.23	0.00	23.91
0.6	Nominal	8.20	16.39	16.39	16.39	16.39	16.39	9.84	-
	Worn surface A	7.38	15.03	11.79	5.97	14.18	20.95	0.00	24.70
	Worn surface B	2.58	4.14	8.03	3.90	34.60	4.11	30.46	12.17
	Debris	2.17	2.36	3.89	6.50	7.06	3.39	0.00	70.09
1.0	Nominal	7.69	15.38	15.38	15.38	15.38	15.38	15.38	-
	Worn surface A	9.07	13.26	13.09	13.79	13.77	12.85	0.00	24.17
	Worn surface B	3.06	3.64	7.97	4.41	36.17	3.62	37.38	3.76
	Debris	0.69	2.55	4.72	0.07	27.51	0.00	0.00	64.47

compounds in most HEAs can be partly explained by the significant contribution of the high entropy (ΔS_{mix}) effect in case that the enthalpy (ΔH_{mix}) is not too negative or positive[15-17]. However, high mixing entropy is insufficient to dominate the formation of phases in HEAs.

Zhang et al. related the formation of simple solid solution to the mixing enthalpy (ΔH_{mix}) and atomic size difference (δ) and subsequently proposed solid-solution formation criteria for HEAs where two parameters, Ω, the entropy of mixing timing the average melting temperature of the elements over the enthalpy of mixing, and δ, the mean square deviation of the atomic size of elements, were used to estimate the phase formation behavior of HEAs[15,18]. Ω and δ are defined by Eqs. (1) and (2), respectively.

$$\Omega = \frac{T_{\text{m}} \Delta S_{\text{mix}}}{\left| \Delta H_{\text{mix}} \right|} \tag{1}$$

$$\delta = \sqrt{\sum_{i=1}^{n} c_i \left(1 - r_i / \bar{r}\right)^2} \tag{2}$$

$$\bar{r} = \sum_{i=1}^{n} c_i r_i$$

where, c_i is the atomic percentage of the ith component, is the average atomic radius and r_i is the atomic radius of the ith component. T_{m}, ΔS_{mix} and ΔH_{mix} are calculated as follows:

$$T_{\text{m}} = \sum_{i=1}^{i=n} c_i \left(T_{\text{m}}\right)_i \tag{3}$$

where, $(T_{\text{m}})_i$ is the melting temperature of the ith component of the alloy.

$$\Delta S_{\text{mix}} = -4R \sum_{i=1, i \neq j}^{n} \Delta H_{ij}^{\text{mix}} c_i c_j \tag{4}$$

where, R is the gas constant (8.314 J/K·mol), c_i and c_j are the atomic percentage of the ith and jth components, respectively, and $\Delta H_{ij}^{\text{mix}}$ is the enthalpy of mixing of binary liquid alloys. Ω represents the competition between $T_{\text{m}}\Delta S_{\text{mix}}$ and ΔH_{mix}, δ represents the atomic size effects. By analyzing the parameters of Ω and δ for various reported HEAs, $\Omega \geq 1.1$ and $\delta \leq 6.6\%$ have been adopted as the criterion for facilitating the formation of a simple solid-solution phase[18]. The values of Ω and δ of the $\text{Al}_{0.5}\text{CoCrCuFeNiB}_x$ alloys with varying boron contents are calculated as shown in Fig.6. In the alloy without boron addition, Ω and δ take values of 16.26 and 4.1%, respectively. Both Ω and δ values satisfy the criterion mentioned here before, and thus, this alloy exhibits superiority in the formation of simple solid solutions. Copper has positive mixing enthalpies with cobalt, chromium and iron; thus, in the alloy without boron addition, copper does not tend to mix with cobalt, chromium and iron but segregates at the interdendrite regions causing its depletion in the matrix fcc phase. The existence of aluminum and nickel at the interdendrite regions is because aluminum and nickel have greater affinity with copper than cobalt, chromium and iron.

In the alloys with boron additions, the value of Ω decreases and that of δ increases with increasing boron content. Ω still satisfies the solid-solution formation criterion, but δ does not. Based on SEM and EPMA analysis, the alloys with boron additions consist of only three or four types of phases which is much fewer than expected. The formation of borides and ordered fcc phase in the alloys with boron additions suggest that not all elements are randomly distributed in the crystal lattice. The entropy of mixing indicates the tendency for the formation of random solution while enthalpy of mixing indicates the tendency for ordering or clustering. $\Omega \geq 1.1$ is obtained in the alloys with boron additions indicating $T_{\text{m}}\Delta S_{\text{mix}}$ is the predominant part of the free energy. The formation of fcc solid-solution phase in the alloys with boron additions is correlated to the alloys' high mixing entropy. The relatively large value of δ indicates that the atomic size difference between components is too large for this alloy system to form entire solid-solution phases. The atomic radius of boron is significantly different from other elements. The large atomic size mismatch would lead to serious lattice distortion and thus increase the corresponding strain energy lowering the stability of solid-solution. Boron has a strong tendency to form borides and the interstitial solubility of boron is quite limited because the mixing enthalpy of the atomic pairs between boron and other elements (except for aluminum and copper which have no borides formation) are highly negative (Table 3). Thus, boron is the main contributor to the negative enthalpy of mixing in the alloys with boron additions.

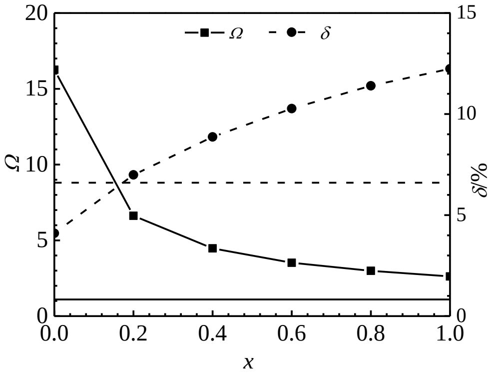

Fig.6 Ω and δ values as functions of boron content in the $\text{Al}_{0.5}\text{CoCrCuFeNiB}_x$ alloys

The variations of the microstructure are responsible for the enhancement of the hardness and the wear resistance. The in-situ formation of borides in the alloys with boron addition results in a composite microstructure, i. e. hard borides within the ductile and tough fcc phase matrix. The microhardness test of the borides indicates that the average hardness HV value is 20470 MPa for the CrB-type boride and 9970 MPa for the (Cr, Fe, Co)B-type boride. Wear mechanism in this multiphase composite mainly depends upon the volume fraction, distribution and morphology of the hard particles[19]. The

Table 3 Chemical mixing enthalpies (ΔH_{mix}) of the possible atom pairs in the $Al_{0.5}CoCrCuFeNiB_x$ alloys (kJ/mol)

Element	B	Al	Co	Cr	Cu	Fe	Ni
B (0.088 nm)	-	-	−24	−31	-	−26	−24
Al (0.143 nm)	-	-	−19	−10	−1	−11	−22
Co (0.126 nm)	-	-	-	−4	6	−1	0
Cr (0.125 nm)	-	-	-	-	12	−1	−7
Cu (0.128 nm)	-	-	-	-	-	13	4
Fe (0.126 nm)	-	-	-	-	-	-	−2
Ni (0.124 nm)	-	-	-	-	-	-	-

enhancement of the wear resistance in the alloys with $x \geq 0.6$ originates from the combination of the large hard borides and relatively ductile and tough fcc matrix. The borides, presenting higher hardness and good high temperature stability can carry the load and resist the plastic deformation due to the friction heat when dry-sliding with the counterpart and thus play a dominant role in resisting abrasive and adhesive wear. The strong resistance of the hard borides to plastic deformation and delamination might keep the oxide layer withstanding the abrasion. The relatively ductile and tough fcc matrix helps supporting the borides and avoiding materials loss by limiting brittle fracture propagation although the work hardening of the fcc phase may be partially responsible for the enhancement of wear resistance. The borides are formed in-situ during the solidification process, so the interfacial bonding tends to be stronger, resulting in the absence of pullout of the borides particles. Also, the large covalent contribution to the chemical bond in borides provides good resistance to metallic adhesion during dry sliding wear [20-22]. Based on the thermodynamics analysis, the simple solid solution phases, such as fcc and/or bcc, can not be guaranteed solely by the high entropy effect. In many cases, intermetallic compounds possibly form in HEAs and much better properties than the HEAs with solely simple solid solution phases could be obtained. From the point of view in developing novel wear resistant materials, HEAs with composited microstructure have great practical potential through the in-situ formation of intermetallic compounds with high hardness and good high-temperature stability within the simple solid solution phase matrix by manipulating the design of alloy compositions.

4 Conclusions

1) Boron precipitates as borides instead of entering the crystal structure of the fcc matrix phase in as-cast $Al_{0.5}CoCrCuFeNiB_x$ alloys. Borides precipitation significantly hardens the alloy and the hardness shows strong correlation to the boron content.

2) There is no distinct difference in the wear resistance of the alloys with $x \leq 0.4$, but the wear resistance shows strong relation to the boron content in the alloys with $x \geq 0.6$. The wear mechanism changes from delamination wear to oxidative wear. The great enhancement in wear resistance is due to the combined effects of the ductile fcc matrix phase and the large hard borides.

3) Composited microstructures in HEAs having excellent wear property could be obtained through the in-situ formation of intermetallic compounds with high hardness and good high-temperature stability within the simple solid solution phase matrix by manipulating the design of alloy compositions.

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B 对 $Al_{0.5}CoCrCuFeNi$ 高熵合金组织、相组成及耐磨性能的影响

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摘　要：研究了 $Al_{0.5}CoCrCuFeNiB_x$($x = 0$~1)的组织、相组成、硬度及耐磨性能。并预测了 $Al_{0.5}CoCrCuFeNiB_x$($x = 0$~1)中简单固溶体形成规律。未添加硼元素的合金具有简单 fcc 固溶体结构。添加硼元素后，合金由简单 fcc 固溶体及多元硼化物组成。硼以硼化物形式析出，没有固溶到 fcc 固溶体中，因而添加硼对 fcc 固溶体的晶格常数无影响。硼化物的析出使合金的硬度提高，并且硬度随着硼含量的增加而呈线性增加。当硼含量 $x\le 0.4$ 时，合金的磨耗阻抗变化不明显，但当硼含量 $x\ge 0.6$ 时，合金的磨耗阻抗随着硼含量增加而呈线性增加。随着硼含量的增加，合金的磨损机制由粘着磨损转变为氧化磨损。合金硬度与耐磨性能的提高是高硬度的粗大硼化物与韧性的 fcc 固溶体基体共同作用的结果。

关键词：高熵合金；硼化物；硬度；耐磨性能

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