EFFECT OF SINTERING TEMPERATURE ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF FeAlCoCrNiTi0.5 ALLOY

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Abstract - In order to study the influence of sintering temperature on the microstructure, section morphology, hardness and friction and wear properties of the alloy, to obtain the optimum sintering temperature for preparing high-entropy alloys. High-entropy alloy powder was manufactured by the mechanical alloying method, and a series of FeAlCoCrNiTi0.5 high-entropy alloy blocks were prepared by inert atmosphere sintering technology. The results showed that when the sintering temperature was 1000°C, the XRD observation of the high-entropy alloy phase structure is a simple body-centered cubic BCC2 phase and a face-centered cubic FCC phase. SEM observation showed that the microstructure is denser, indicating that the alloy forms a relatively stable structural phase. At this time, the hardness and friction and wear properties of the alloy are the best.

Keywords: High entropy alloy; Microstructure; Sintering temperature; Mechanical properties.

1. Introduction

The high-entropy alloy (HEA) have broad prospects for industrial applications due to their properties such as high strength and toughness [1], high hardness [2-3], wear resistance [4], and excellent resistance to temper softening [5]. However, high-entropy alloys have not yet been extended from the laboratory to the market, that's why a large number of researchers are working in this direction, hoping to promote potential applications in ultra-light materials such as aerospace materials.

HEAs have a large number of obstacles on the road of their research. The current main concern is to improve their structural properties and application, and their preparation process and the post-treatment process have an extremely important impact on their structural properties. Sintering produces a stronger metallurgical bond between the powder particles. The process parameters include the sintering temperature. Since the sintering temperature directly affects the microstructure of the ingot, including grain size, pore size, grain boundary shape and distribution [6], the sintering temperature is changed to observe the performance change of the HEA in this study.

AlFeNiCoCr series high entropy alloys [7] have been widely studied. Recently reported AlCoCrFeNiTi0.5 high entropy alloys have outstanding comprehensive mechanical properties in the explored high-entropy alloy systems and can be on a par with most of the high-strength alloys even bulk amorphous alloys are comparable that have been publicly reported [8]. Although a lot of researches have been done on high-entropy alloys, the research on high-entropy alloys prepared by mechanical alloying and the research on heat treatment processes on the high-entropy alloys are relatively rare. Therefore, this study used mechanical alloying combined with vacuum sintering technology to prepare FeAlCoCrNiTi0.5 high entropy alloy, change its sintering temperature, study the mechanical properties and microstructure of the alloy. At the same time, the influences of different sintering processes on the microstructure, hardness, friction and wear properties and the mechanism of action of the alloys are explored in order to provide the necessary data accumulation and technical support for the engineering application of these alloys.

2. Materials and Methods

In order to study the effect of post-sintering temperature on the microstructure and mechanical properties of high-entropy alloy FeAlCoCrNiTi0.5, FeAlCoCrNiTi0.5 alloy powder was prepared by the mechanical alloying method. The molar ratio of Fe, Al, Co, Cr, Ni, Ti elements was 1:1:1:1:1:0.5.

The electronic molecular balance (ME204E type) with an accuracy of 0.01g was weighed, and the weighed metal powder was placed in a variable frequency planetary ball mill (XQM-0.4L type) for mixing. The ball mill used a vacuum stainless steel tank and a stainless steel ball with a ball-to-batch ratio of 10:1, a ball milling time of 20 h, and a rotational speed of 350 r/min. After alloying the metal powder, weigh 5g of alloy powder, add 0.2g of stearic acid as a binder, put it into the mortar and grind it evenly, and press the powder into tablets with a tableting machine (DY-30 type), the pressure was 35kpa. Hold the pressure for 30 min to obtain a block sample (Figure 1).

The bulk sample was placed in a tube furnace in an inert gas atmosphere and sintered at 700 ° C, 800 ° C, 900 ° C, and 1000 ° C. After sintering, the samples were tested. The phase structure of the high-entropy alloy was observed by XRD, and then the surface hardness of the samples was measured using a microhardness tester. The friction and wear properties of the samples were tested using a material surface performance tester. Finally, the microstructure of the high-entropy alloy was examined by SEM.

Figure 1: Specimen

3. Results and Discussion

3.1 Phase Analysis

Figure 2 shows the X-ray diffraction spectrum of high-entropy alloy AlFeCoCrNiTi0.5 at different sintering temperatures. We can know that the high-entropy alloy AlFeCoCrNiTi0.5 is mainly composed of two body-centered cubic phase, BCC1 phase and BCC2 phase through analysis. This is also in line with the core effect of high-entropy alloys, namely the high entropy effect. It refers to the thermodynamically hindered formation of complex phases. Because of the traditional metallurgical laws, multi-element metals tend to form a variety of compounds and segregation phases due to the different interactions between the elements, and the resulting structure is not only complicated but difficult to analyze and brittle. However, the high entropy effect can inhibit the formation of various intermetallic compounds, making the high-entropy alloy exhibit a simple body-centered cubic structure or a face-centered cubic structure [9]. At the same time, the atomic size difference in the high-entropy alloy AlFeCoCrNiTi0.5 is large, and the degree of lattice distortion is serious, so it is more inclined to form a loose body-centered cubic phase, thereby adjusting the strain on the lattice to reduce the free energy of the system. According to the correspondence of the diffraction peaks in the diffraction pattern, it can be found that BCC1 was correspondingly enriched in Al-Ni phase, while BCC2 was correspondingly enriched in Fe-Cr phase. This conclusion can also be confirmed in previous studies [10].

It also can be seen from the above four figures that when the sintering temperature is 700 °C, the content of BCC1 phase in the high-entropy alloy is dominant, much larger than the content of BCC2. As the temperature rises to 800 °C, the BCC1 phase is still the main phase in the alloy, but the intensity ratio of the peak in the diffraction pattern shows that the content of BCC1 is decreased, and the content of BCC2 is gradually increasing. Continue to increase the sintering temperature, and found that not only the two individual body-centered cubic phases of BCC1 and BCC2 were detected in the XRD detection range, but also the existence of the ω phase was detected. By comparing the PDF standard card, it is found that the angle and peak of the ω phase diffraction peak correspond to the diffraction peak of AlTi3.When the sintering temperature is raised to 1000 ° C, the presence of the BCC1 phase has not been detected by the detection accuracy range of XRD. In order to quantitatively compare the conversion ratio of the main phase in the high-entropy alloy, we determine the total contribution of the phase as the sum of the volume fractions of the main phases BCC1 and BCC2, regardless of the presence of other phases, considering only the total integrated intensity of all observed diffraction peaks of the main phase. Table 1 gives the volume fractions of the main phases BCC1 and BCC2 at different sintering temperatures. It can be clearly seen that gradually increasing the sintering temperature will facilitate the phase transition from BCC1 to BCC2.According to the angle of the diffraction peak combined with the Bragg equation, the size of the unit cell in the crystal structure can be calculated, the lattice parameters of each main phase in the alloy are given in Table 2. It can be seen that the body-centered cubic phase BCC2 has a larger lattice constant and a looser structure, so the above transition occurs in the alloy, which is consistent with the conclusions of our previous analysis.

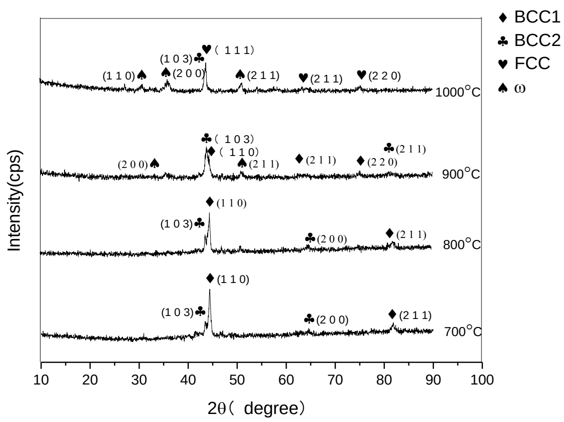

Figure 2: XRD Image of AlCoCrFeNiTi0.5 at Different Sintering Temperatures

Table 1. Percentage of volume fraction of the main phase for the different sintering temperature

V.fraction	700(2h)	800(2h)	900(2h)	1000(2h)
V_{bcc1}[%]	63.61	61.08	51.74	0
V_{bcc2}[%]	36.39	38.92	48.25	100

Table 2. The cell parameters of high entropy alloys at different sintering temperatures

Sintering temperature（℃）	**BCC1/Å**	**BCC2/ Å**	**ω-phase/ Å**
700(2h)	2.8843	6.5756	-
800(2h)	2.8791	6.5296	-
900(2h)	2.8649	6.5028	2.4634/4.0236
1000(2h)	-	6.5605	2.3440/3.8284

3.2 Micro Morphology Analysis

In this study, the fracture morphology of high-entropy alloys with different compositions was observed by SEM to analyze the micro-morphology of high-entropy alloys. Figure 3 is a SEM micro-morphology of a FeAlCoCrNiTi0.5 high-entropy alloy sintered at 700 ° C, 800 ° C, 900 ° C, and 1000 ° C in an inert atmosphere. It can be clearly seen from Fig. 2 that the macrostructure of the high-entropy alloy after sintering is equiaxed. We can also observe that the microstructure of the alloy is denser after sintering, and a nanocrystalline phase is produced.

This may be because, during the sintering process, the alloy undergoes crystal deformation and phase transformation, resulting in a shear stress in some directions in the crystal lattice. When the stress exceeds the critical shear stress of a particular twin system, the twin system is activated, and some specific crystal planes in some grains will be relatively slipped, resulting in deformation twins [1].

But at the same time, it can be found that there are still some pores in the sintered block.

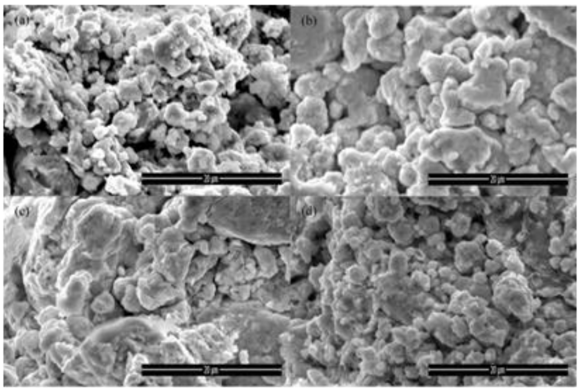

Figure 3: SEM photographs of 10000-fold magnification of samples incubated at 700°C, 800°C, 900°C, and 1000°C

When the maximum temperature is 700 ° C, the alloy has less bulk structure, the fracture shows different degrees of grain polyhedron shape, the graininess is obvious, and there are still many holes because the inside of the grain in the alloy is mostly composed of amplitude modulation structure [11].

When the temperature rises to 800 ° C and 900 ° C, the graininess of the alloy is significantly reduced, the porosity is lowered, the agglomeration phenomenon in the alloy is obvious, the density is increased, and the crystal grains are gradually refined because the elevated temperature increases the diffusion efficiency of the element and makes the alloying elements more uniform. However, when the sintering temperature rose to 1000 ° C, we observed that the grains were continuously roughened.

This is because as the heat treatment temperature is further increased, the grain size of the alloy is continuously increased, but the degree of growth is small, the atomic diffusion coefficient is large, and the grain boundaries are more likely to migrate, So the crystal coarsening becomes larger. But at 1000 °C we can clearly observe the appearance of a network in the alloy. Here we believe that the nanocrystalline phase that occurs at lower sintering temperatures is interwoven at high temperatures, resulting in a more stable phase in the alloy.

3.3 Hardness Analysis

The addition of large atomic radius elements such as Al and Ti to the high-entropy alloy enhances lattice distortion and solid solution strengthening, which makes the high-entropy alloy have a higher hardness. Secondly, through mechanical alloying, the HEA can obtain finer crystal grains than the casting conditions. The ball milling process causes strong lattice distortion and forms a supersaturated solid solution. In the sintering process, phase transformation often occurs to produce nanometers.

Precipitating phases, these make the strengthening effect more obvious.

The measurement results are shown in Figure 4.

As can be seen from Fig. 4, as the holding temperature increases, the hardness gradually increases, and the hardness is maximum at 1000 ° C, reaching 518.67 HV. This is because as the sintering temperature increases, the phase structure of the high-entropy alloy continuously changes from BCC1 phase to BCC2 phase, and X-ray diffraction pattern analysis confirms that BCC1 is correspondingly enriched in Al-Ni phase, while BCC2 is relatively enriched in Fe-Cr phase, compared with Fe-Cr solid solution has higher hardness and stability, this conclusion has also been verified in previous studies [12], so the hardness of high-entropy alloy is increasing, while at sintering temperature After reaching 900 °C, the presence of the intermetallic compound AlTi3 was detected in the high-entropy alloy AlCoCrFeNiTi0.5. The covalent bond in the AlTi3 phase can ensure the alloy has higher hardness, so the hardness of the high-entropy alloy is further improved after the temperature is raised.

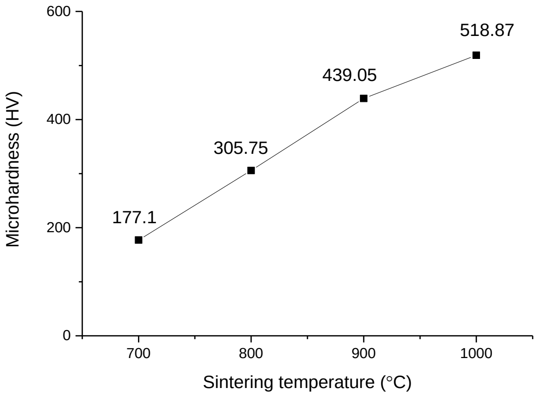

Figure 4: Microhardness values of specimens incubated for 2 hours at different sintering temperatures

3.4 Friction and Wear Analysis

Figure 5 shows the measured friction and wear coefficient and Figure 6 shows the measured wear.

The friction coefficient is an important parameter used to indicate the friction properties of materials.

Therefore, the friction and wear properties of high-entropy alloys after vacuum hot-pressing sintering are studied, and the effects of different sintering processes on the wear resistance of high-entropy alloy FeAlCoCrNiTi0.5 are discussed. The smaller the coefficient of friction is, the less the amount of wear, indicating the better the friction properties of the material.

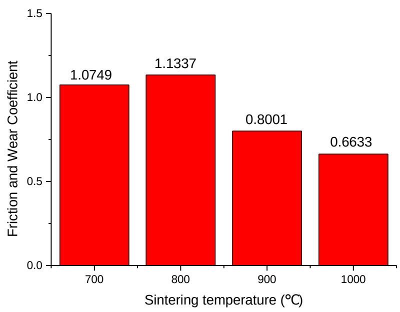

Figure 5: Friction and Wear Coefficient of AlCoCrFeNiTi0.5 at Different Sintering Temperatures

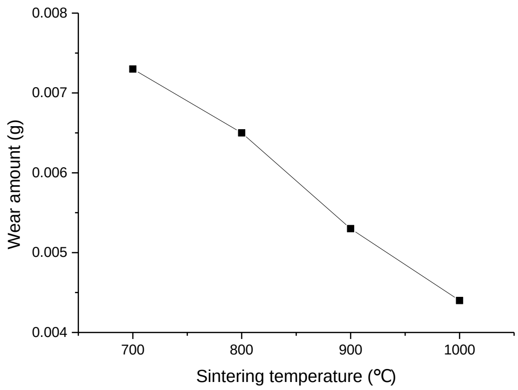

Figure 6: The wear of different sintering temperature specimens

It can be seen in Fig. 5and Fig. 6 that the high-entropy alloy exhibits excellent friction and wear properties, and the friction coefficient is relatively stable in the friction test process. As the sintering temperature increases, the average friction coefficient decreases overalland the amount of wear gradually decreases. When the sintering temperature is low, the structure is not dense enough, and there are many pores, so the friction coefficient is high and the wear amount is also large.

As the sintering temperature increases, some intermetallic compounds or other crystal phases melt and infiltrate to fill some of the pores in the high-entropy alloy, which makes the structure denser and the surface is flatter, so the friction coefficient is reduced and the wear amount is reduced. The comprehensive analysis found that the FeAlCoCrNiTi0.5 high-entropy alloy had the best friction and wear properties after 1000 °C sintering.

4. Conclusions

(1) The XRD results show that the high-entropy alloy AlFeCoCrNiTi0.5 is mainly composed of body-centered cubic BCC1 and BCC2, and the main phase in the alloy gradually changes from BCC1 to BCC2 as the sintering temperature increases. The presence of the intermetallic compound AlTi3 appeared at 900 °C.

(2) The high-entropy alloy AlFeCoCrNiTi0.5 is a porous structure. The sintered bulk alloy has a denser structure. And as the sintering temperature increases, a more stable phase will gradually form in the alloy, and a network structure can be observed in the alloy at 1000 °C.

(3) In the measurement of microhardness and friction and wear experiments, the sintering temperature is positively correlated with the performance of the metal. The higher the sintering temperature is, the higher the hardness of this alloy is and the lower the average friction coefficient is.

Based on the above conclusions, it is known that increasing the sintering temperature is beneficial to atomic diffusion, forming a more stable structural phase in the alloy, and enhancing various comprehensive properties of the high-entropy alloy.
Competing Interests: The authors declare that there are no competing interests regarding the publication of this paper.

Acknowledgments

The research is funded partially by the Fundamental Research Funds for the Central Universities fund (KYZ201760) and the Open topic of Jiangsu Provincial Key Laboratory of Advanced Manufacturing Technology (HGAMTL-1711)

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