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A Review of Preparation Methods and Strengthening Mechanisms of AlCoCrFeNi-Based High-Entropy Alloys

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ABSTRACT

This paper reviews the development, preparation methods, and strengthening mechanisms of high-entropy alloys (HEAs). Different from traditional alloys, HEAs contain at least five principal elements and show severe lattice distortion and chemical short-range order. These features give HEAs special mechanical properties. The article first introduces the background and research status of HEAs, especially the AlCoCrFeNi system. Then, it summarizes common preparation methods, such as vacuum melting, powder metallurgy, laser cladding, and high-temperature high-pressure sintering. After that, it discusses five main strengthening mechanisms: second-phase strengthening, work hardening, grain refinement, heterostructure strengthening, and solid-solution strengthening (substitutional and interstitial). Finally, it points out that the interstitial strengthening mechanism is still unclear, and more work is needed on composition design, heat treatment, and the cooperation among different strengthening methods. Further study on HEAs will provide new ideas for designing high-performance materials.

KEYWORDS

High-entropy alloy; Preparation method; Strengthening mechanism; Microstructure

1. INTRODUCTION

1.1. Background and Importance of High-Entropy Alloys

Since the early 20th century, traditional alloy design has been based on one main element plus a few alloying additions to reach a single target property. This idea is now limited because the number of principal elements is small, and property improvement has almost stopped. Modern industry needs higher performance, and conventional alloys can no longer satisfy these needs. At the end of the 20th century, researchers noticed that some alloys with very high mixing entropy can form a single-phase solid solution. Therefore, a new design concept called high-entropy alloys (HEAs) appeared. In HEAs, at least five principal elements are mixed, and every element has an atomic fraction between 5 % and 35 %. Ye and co-workers first reported this idea in 2004. They prepared equiatomic alloys containing five or more metals and found special properties. The phases in HEAs are usually face-centered cubic (FCC), body-centered cubic (BCC), hexagonal close-packed (HCP), or a mixture of these structures. Each phase shows different mechanical properties, so the required phase can be obtained by adjusting the element ratio to fit different service conditions. The HEA concept breaks the limits of traditional alloy composition, structure, and performance. The word “entropy” comes from the multi-element composition. High mixing entropy helps the alloy form a single solid solution or complex phases and produces new physical, chemical, and mechanical properties

1.2. Research Status of AlCoCrFeNi High-Entropy Alloys

The unique microstructure, good properties, and wide application potential of HEAs have attracted many researchers since the idea was proposed. Because every principal element can act as a solute, the difference in atomic size among the elements produces severe lattice distortion. Among many HEA systems, AlCoCrFeNi has received much attention because it contains light Al and several transition metals (Co, Cr, Fe, Ni). The light Al decreases density, and the transition metals improve overall mechanical performance through multi-element synergy. Up to now, Fe, Ni, Co, Cr, and Al are the five most studied elements in HEAs. AlCoCrFeNi alloys show good combination of strength and moderate resistance to high-speed shear, so they are used in aerospace, crash boxes, and armor materials. These alloys are usually prepared by vacuum arc melting, although spark plasma sintering is also reported. However, spark plasma sintering needs strict conditions, so arc melting is still the main method in practice.

Research on HEAs is still at an early stage. Some studies show that accurate control of element ratio can increase hardness, wear resistance, and fatigue resistance. Besides element interaction, the influence of processing route and strengthening mechanism on microstructure and overall properties needs deeper study. Because HEAs show great potential and wide application, this paper systematically reviews preparation methods and strengthening mechanisms and provides guidance for future design and processing, so that these alloys can be used in engineering more quickly.

2. PREPARATION METHODS OF HIGH-ENTROPY ALLOYS

The earliest methods were vacuum arc melting and vacuum induction melting. Later, mechanical alloying, powder metallurgy, and laser cladding were developed. Many industrial and functional applications need both high strength and good ductility. Traditional processing often sacrifices ductility to obtain high strength, while high-pressure treatment can overcome this trade-off. Alloys prepared under high pressure show high strength and large uniform elongation without interface-induced embrittlement or plastic instability often seen in eutectic HEAs. In this section, conventional methods and high-temperature high-pressure sintering are briefly introduced.

2.1. Vacuum melting methods

Most researchers now use vacuum melting. The methods include vacuum arc melting, vacuum induction melting, and vacuum electron beam melting. The reaction temperature is high, so volatile impurities can evaporate without damaging the alloy. Repeated melting can give uniform ingots suitable for laboratory study. Karati et al. used vacuum arc melting to prepare AlCoFeMnNi and studied thermal stability and phase evolution. Cai et al. used vacuum induction melting to prepare FeCoCrNiMn and studied tensile behavior and microstructure evolution after uniaxial quasi-static loading. These methods reach high temperature and can melt high-melting-point alloys while removing volatile impurities and gases. However, shrinkage, segregation, porosity, and high internal stress often appear during solidification, and these defects influence properties.

2.2. Powder metallurgy

Powder metallurgy uses metal or non-metal powders. After cold pressing and sintering, the powders form a dense body. During pressing at room temperature, powder particles undergo mechanical interlocking, plastic deformation, and work hardening. During sintering, diffusion, welding, chemical reaction, dissolution, and recrystallization replace mechanical bonding with metallurgical bonding, and a strong bulk is formed. The advantages are low sintering temperature, no segregation, high material utilization (above 90 %), and low cost for large-scale production. The main disadvantage is that the strength of the product is slightly lower than that prepared by melting, so this route is seldom used for laboratory study.

2.3. Laser cladding

Laser cladding concentrates heat in a small zone, so the process time is short and the cooling rate is high. Fast cooling promotes nucleation of the solid solution and avoids unwanted intermetallics. Li used selective laser melting to study CoCrFeMnNi powder and analyzed non-equilibrium microstructure evolution and mechanical properties. Zhang used laser cladding to prepare FeCrNiCoMnBx coating and studied structure and properties. However, fast cooling easily produces lack of fusion, unmelted particles, shape defects, cracks, and pores, especially when the melting point is high and oxidation resistance is poor.

2.4. Electrochemical deposition

Electrochemical deposition can prepare thin films, but the film often contains defects that influence mechanical and electrical properties and decrease quality. Yao used this method to study magnetic properties of nanostructured amorphous FeCoNiMnNd, but the method is still seldom used in experiment or industry because of its own limits.

2.5. High-temperature high-pressure sintering

Song found that when sintering pressure was below 800 MPa, increasing pressure decreased the sintering temperature needed to reach the same density for nano-cubic zirconia. Yao reported that high-pressure high-temperature treatment can overcome the strength-ductility trade-off. A brittle eutectic HEA was treated under high pressure; its strength doubled to 1150 MPa, and tensile ductility increased by 36 %. High-pressure-induced microstructure relieved stress concentration at interfaces and stopped interface cracking common in conventional eutectic high alloys. These results point to a new direction for developing next-generation structural materials. The combination of pressure and temperature offers a wide window to tailor microstructure and to overcome the strength-ductility or strength-conductivity trade-off.

Besides the above methods, high-velocity oxy-fuel spraying for HEA thin films and melt spinning for amorphous HEA ribbons have also been reported. Traditional casting, forging, and coating can also be used to prepare bulk, coating, and film products.

3. STRENGTHENING MECHANISMS OF HIGH-ENTROPY ALLOYS

In general, the strength of HEAs is much higher than that of conventional solid-solution alloys. In HEAs, every principal element has a high fraction, so there is no clear solvent-solute difference, and the solid-solubility is high. Differences in atomic radius and modulus among the elements produce severe lattice distortion and a stress field that blocks dislocation motion. With suitable alloy design, the strength can be further increased. Common strengthening routes in HEAs include work hardening, grain refinement, interstitial doping, second-phase strengthening, and heterostructure design. The theory of strengthening is a hot topic in HEA research.

3.1. Second-phase Strengthening

Among HEAs, FCC alloys are most studied, BCC alloys are less studied, and HCP alloys are seldom reported. FCC alloys show good ductility but low strength. Introducing a second phase is now an important route to increase strength. By suitable matrix design and alloying, many second phases (L12, μ, η, L21, B2, σ, etc.) can form. These phases increase strength and keep reasonable ductility.

Second-phase strengthening includes precipitation strengthening and dispersion strengthening obtained by heat treatment or internal oxidation.

Otto found that when the grain size of FeCoNiCrMn was reduced from 155 µm to 4.4 µm, the tensile strength increased from 520 MPa to 670 MPa, but elongation decreased from 80 % to 60 %. Even after grain-boundary strengthening, the strength was still lower than that of advanced steels such as TRIP steel (~800 MPa). Hadraba added 3 % (mass fraction) Y2O3 nanoparticles into CoCrFeMnNi. At both room temperature and 800 °C, the yield strength of the nanoparticle-reinforced alloy was at least 300 MPa higher than that of the single-phase alloy, but ductility decreased by at least 50 %. Compressive yield strength was about 200 MPa higher, and plasticity was still good.

3.2. Work Hardening

Work hardening means that plastic deformation below the recrystallization temperature increases strength and hardness but decreases ductility. The mechanism is that plastic deformation produces a high density of dislocations; interaction among dislocations causes pile-up and increases the resistance to dislocation motion, so strength increases. Usually, the hardening effect is proportional to the square root of dislocation density; larger deformation gives higher strength. Common methods are cold rolling, cold forging, and cold drawing.

In HEAs, work hardening is mainly used for single-phase FCC alloys that have good deformation ability; it is seldom used for BCC or multiphase alloys with poor deformability. Stepanov cold-rolled FeCoNiCrMn to a true strain of 80 % at room temperature and at liquid nitrogen temperature. The ultimate tensile strength reached 1.5 GPa and 1.2 GPa, respectively, but elongation dropped sharply from ~70 % to ~20 %. The high strength obtained by cold deformation is easily lost after recovery or recrystallization heat treatment.

3.3. Grain Refinement Strengthening

During deformation, grain boundaries can block dislocations and increase yield strength. Grain refinement increases the density of grain boundaries and gives strong strengthening; it can even increase strength and keep good ductility. Grain refinement follows the Hall–Petch relation: smaller grain size gives higher yield strength. Grain refinement is effective for HEAs, especially for single-phase FCC HEAs, and is one of the main strengthening methods. Because of the unique chemical and structural features of HEAs, grain refinement shows new characteristics.

Wu cold-rolled and annealed as-cast Al0.1CoCrFeNi; grain size decreased from 115 µm to 7.9 µm, and yield strength increased from 119 MPa (as-cast) to 366 MPa (annealed) while good tensile plasticity was kept. Hou studied the relation between grain size and yield strength in dual-phase Al0.45CoCrFeNi and found that the strengthening effect is the sum of the contribution from each phase and from the unrecrystallized matrix. For eutectic AlCoCrFeNi2.1, thermo-mechanical treatment also gave grain refinement; after annealing, yield strength increased from 620 MPa (as-cast lamellar) to 1 100 MPa (fine-grained). These results show that grain size control is effective for both single-phase FCC and dual-phase FCC+BCC HEAs.

3.4. Heterostructure Strengthening

Under back stress, the soft region can become almost as strong as the hard region, so the overall yield strength increases. In the later stage, both soft and hard regions undergo plastic deformation; the soft region carries more plastic strain and strain partitioning appears. When soft and hard regions carry different strains, a strain gradient exists near the interface. The gradient increases with strain partitioning and produces back-stress work hardening that delays necking and increases uniform elongation.

Therefore, intentionally introducing heterostructures such as gradient structure, partial recrystallization, bimodal grain distribution, or heterogeneous lamellae can promote heterogeneous plastic deformation, increase strain-hardening ability, and enhance uniform tensile ductility.

3.5. Solid-solution Strengthening

Solid-solution strengthening is a main route in HEAs. Every principal element has a high fraction, so the lattice sites are occupied by different atoms. Although the atomic radii are close, the high mixing level still produces a strong lattice distortion. Up to now, no quantitative method exists to describe this distortion. Because of the large lattice distortion, the classical Burgers vector may no longer be a fixed value but may become a distribution. This uncertainty brings difficulty to theoretical calculation and to understanding deformation and strengthening mechanisms. Compared with conventional single-principal-element solid solutions, the strengthening effect in multi-principal-element HEAs is more complex. Solid-solution strengthening in HEAs includes substitutional and interstitial routes.

3.5.1. Substitutional solid-solution strengthening

In substitutional solid solutions, solute atoms occupy lattice sites of the solvent. Differences in atomic size produce lattice distortion. In HEAs, every element is present at a high level, so the concepts of solute and solvent are not clear. Because the atomic radii of the elements are different, HEAs themselves are supersaturated solid solutions with large lattice distortion. Adding extra elements with large radius difference increases distortion and produces obvious strengthening.

He studied the effect of Al on tensile properties of FeCoNiCrMnAlx. Al produced strong solid-solution strengthening and induced BCC phase precipitation. With increasing Al, hardness and yield strength continuously increased. Zhou studied the effect of Ti on compressive properties of AlCoCrFeNiTix ($x = 0, 0.5, 1, 1.5$). When $x = 0$, yield strength was 1.5 GPa; when $x = 1.5$, it increased to 2.22 GPa. Because the atomic radius of Ti is larger than that of the other elements, Ti occupies lattice sites and produces stronger lattice distortion and higher distortion energy, so yield strength increases obviously.

3.5.2. Interstitial solid-solution strengthening

Small atoms such as C, N, and B can enter the lattice and give interstitial solid-solution strengthening. Based on present studies, the effects of these four interstitial atoms (C, N, B) on microstructure and properties are summarized. Understanding the role of interstitial atoms helps to design microstructure and to meet different property requirements.

(1) C interstitial strengthening

C is a common alloying element. In austenite, C has high solubility and can form a solid solution to increase strength; it can also form carbides to increase hardness and wear resistance. Stepanov added 1 at % C into CoCrFeNiMn. The results show that C increases dislocation activity and delays deformation twinning. At the same grain size, the average yield strength of CoCrFeNiMn-1at%C is 200 MPa higher than that of the C-free alloy, so the strengthening effect is clear.

(2) N interstitial strengthening

N is often added in conventional alloys. N has an atomic size similar to C and can produce strong strengthening. N doping gives both interstitial and solid-solution strengthening and improves strength, ductility, and wear resistance. Zhang prepared Ti2ZrHfV0.5Mo0.2 and studied the effect of N content. With increasing N, the structure changed from single BCC to BCC plus nitrides. When N reached 4 at %, hardness increased by ~73 % (346 HV → 598 HV) and compressive yield strength increased by ~80 % (1 026 MPa → 1 849 MPa), but plasticity decreased sharply. The alloy with 1 at % N showed the best balance: yield strength 1 193 MPa and plastic strain >50 %. N gives solid-solution strengthening and increases strength and hardness while keeping plasticity almost unchanged; when N content is too high, nitride volume fraction and size increase, and ductility and toughness decrease.

(3) B interstitial strengthening

B has a low enthalpy of formation and easily forms hard second phases; it can also refine grains and strengthen grain boundaries, so yield strength increases obviously. Seol added trace B into Fe20Mn20Cr20Co20Ni20. With only 30 μg/g B, yield strength increased by more than 100 % and tensile strength increased by ~40 %, with only a small loss in plasticity. Trace B gives interstitial solid-solution strengthening, grain refinement, and grain-boundary strengthening. After B addition, strain-hardening ability also increased. The complex composition and structure of HEAs do not prevent small B atoms from segregating at grain boundaries.

Research on interstitial solid-solution strengthening is still limited. If the doping level is too high, brittle ceramic phases can form and degrade properties. Although interstitial atoms increase strength, they can also produce local stress concentration and cause early failure under external stress, so the interstitial mechanism needs further study.

4. CONCLUSION

Unlike traditional metals, high-entropy alloys (HEAs) show obvious chemical short-range order and heavy lattice distortion. These two features make every strengthening and toughening method behave differently from that in conventional alloys. Although some results are still under discussion, HEAs have great potential for further development and application as a new class of metallic materials. Up to now, remarkable progress has been made in property research, but many questions remain open. For example, the mechanism of interstitial solid-solution strengthening is still incomplete, and both theory and processing routes are lacking. Precise control of phase structure also needs deeper study, including more accurate composition design and better heat-treatment parameters to obtain stable and uniform phases. In addition, the balance among different properties has to be considered. To further improve mechanical properties and to adjust microstructural evolution, more work is required on preparation technology, strengthening methods, and the cooperative effect among different routes. A reasonable combination of these factors can give full play to the material potential. Continued research and development on HEAs will provide a wide design idea for high-performance materials.

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