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Microstructure and properties of age-hardenable Al_xCrFe_{1.5}MnNi_{0.5} alloys

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ABSTRACT

Two promising high-entropy alloys (HEAs) $Al_x CrFe_{1.5} MnNi_{0.5}$ (x=0.3 and 0.5) were designed from Al–Co–Cr–Cu–Fe–Ni alloys by substituting Mn for expensive Co and excluding Cu to avoid Cu segregation. Microstructures and properties were investigated and compared at different states: as-cast, as-homogenized, as-rolled and as-aged states. $Al_{0.3} CrFe_{1.5} MnNi_{0.5}$ alloy in the as-cast, as-homogenized and as-rolled states has a dual-phase structure of BCC phase and FCC phase, in which Al, Ni-rich precipitates of B2-type BCC structure disperse in the BCC phase. $Al_{0.5} CrFe_{1.5} MnNi_{0.5}$ alloy in the corresponding states has a matrix of BCC phase in which Cr-rich particles of BCC structure and Al, Ni-rich precipitates of B2-type BCC structure disperse. These three BCC phases have the same lattice constant.

Both alloys are workable and show a hardness range of Hv 300–500 in the as-cast, as-forged, as-homogenized and as-rolled states. $Al_{0.5}CrFe_{1.5}MnNi_{0.5}$ alloy has a higher hardness level than $Al_{0.3}CrFe_{1.5}MnNi_{0.5}$ one because of its full BCC phase. Both alloys thus can be used as structural parts requiring stronger strength.

Both alloys display a significant high-temperature age-hardening phenomenon. As-cast Al_{0.3}CrFe_{1.5}MnNi_{0.5} alloy can attain the highest hardness, Hv 850, at 600 °C for 100 h, and Al_{0.5}CrFe_{1.5}MnNi_{0.5} can get even higher hardness, Hv 890. The aging hardening is resulted from the formation of ρ phase (Cr₅Fe₆Mn₈-like phase). Prior rolling on the alloys before aging could significantly enhance the age-hardening rate and hardness level due to introduced defects. Al_{0.5}CrFe_{1.5}MnNi_{0.5} alloy exhibits excellent oxidation resistance up to 800 °C, which is better than Al_{0.5}CrFe_{1.5}MnNi_{0.5} alloy. Combining this merit with its high softening resistance and wear resistance as compared to commercial alloys Al_{0.5}CrFe_{1.5}MnNi_{0.5} alloy has the potential for high-temperature structural applications.

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1. Introduction

Almost all conventional alloys have been designed to have a major element, such as ferrous, aluminum, copper, titanium, and magnesium alloys. To extend this conventional concept of alloy designing, a new alloy field of multicomponent compositions was proposed in 1996, which has been coined as "high-entropy alloys (HEAs)". A high-entropy alloy has been suggested to contain at least five principal metal elements, each of which has the content more than 5 at% but less than 35 at% [1,2]. Such alloys are featured with significantly higher mixing entropy than conventional alloys as compared at the liquid solution or regular solid solution state.

Based on the understanding and experience in metallurgy, an alloy with multi-principal elements was thought to be complicated in microstructure and difficult to be analyzed because many intermetallic compounds would form. They would be also brittle to be applied as structural materials [2,3]. However, with suitable alloy design, high-entropy alloys might exhibit unique features, such as (1) forming simple solid solution phases with nano- or even amorphous structure, (2) good thermal stability, (3) high hardness, and (4) superior corrosion and oxidation resistance [1,2,4–26]. They are expected to be abundant in academic research and industrial applications. Four core effects in high-entropy alloys would be more pronounced than those in conventional alloys [27]: (1) high entropy - it enhances the formation of simple solid solution phases, such as FCC and/or BCC structures, and thus simplifies the microstructures; (2) severe lattice distortion – it arises from the atomic size difference among different elements in solid solution phases and might markedly influence mechanical, physical and chemical properties; (3) sluggish diffusion - it is due to the inefficient cooperative diffusion of various species and thus slow down phase transformations; and (4) cocktail - it comes from the ideal mixing and inevitably excess interactions among the multi-principal elements in solid solution phases, and thus is a composite effect on properties.

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Table 1

Different processes applied for Al_xCrFe_{1.5}MnNi_{0.5} HEAs.

States	Processes
As-cast	Arc melting and casting
As-forged	Arc melting and casting → forging at 1200 °C with a thickness reduction 50%
As-homogenized	Arc melting and casting \rightarrow forging at 1200 °C with a thickness reduction
	$50\% \rightarrow homogenizing$ at 1100°C for 6 h
	followed by air cooling
As-rolled	Arc melting and casting \rightarrow forging at 1200 °C
	with a thickness reduction
	$50\% \! \rightarrow \! homogenizing$ at 1100 $^{\circ}C$ for 6 h
	followed by air cooling \rightarrow warm rolling at
	300 °C with 10%, 30%, 50%, and 80% reductions,
	respectively
As-aged	As-cast or as-rolled samples \rightarrow aging at 600 °C,
	700 °C, 800 °C, and 900 °C for 2 h, 5 h, 10 h, 20 h,
	50 h, and 100 h, respectively

Previous studies on the Al_{0.5}CoCrCuFeNi alloy demonstrated that it was of simple FCC phase and exhibits good hot and cold workability [9,10,20,24,26]. It displays high strength and high work-hardening capability up to 800 °C. However, its initial strength level is quite low, as an example, Al_{0.5}CoCrCuFeNi, has the as-cast hardness about 200 Hv which is similar to that of 304 and 316 stainless steels. Moreover, cobalt is an expensive and strategic material which might limit their broad applications.

This study is intended to develop a cheaper alloy system having higher strength and resistance to annealing softening. It was designed to be $Al_xCrFe_{1.5}MnNi_{0.5}$ (x=0.3 and 0.5) alloy based on Al-Co-Cr-Cu-Fe-Ni alloys by substituting Co with Mn referring to the fact that Mn brings good merits for austenite manganese steels [28], and excluding Cu to avoid the Cu segregation in the interdendrite regions [9,10]. In addition, the Fe content is increased so as to further reduce the cost. Two Al contents were chosen for this study because they exhibit pronounced high-temperature aging hardening effect as compared with those Al contents lower than 0.3 or higher than 0.5. These two alloys were investigated on various states: as-cast, as-forged, as-homogenized, as-rolled and as-aged states. The results show that the new alloy system has its uniqueness and the potential in applications.

2. Experimental procedure

The Al_xCrFe_{1.5}MnNi_{0.5} alloys (x = 0.3 and 0.5) were prepared by arc melting elemental Al, Cr, Fe, Mn, and Ni raw materials at a current of 500 A in a water-cooled copper hearth. The source materials had a purity level higher than 99.0 wt%. Melting was performed at a pressure of 0.01 atm after purged with argon three times. Repeated melting for at least four times was done to improve chemical homogeneity of the alloys. The ingots were approximately 30 mm in diameter and 10 mm in thickness and 50 g in weight. First, they were forged with a thickness reduction of 50% at 1200 °C and homogenized at 1100 °C for 6 h followed by air cooling. Then, they were warm rolled at 300 °C with 10%, 30%, 50%, and 80% reductions, respectively. The as-cast and as-rolled samples were annealed at 600 °C, 700 °C, 800 °C, and 900 °C for 2 h, 5 h, 10 h, 20 h, 50 h, and 100 h, respectively. Table 1 summarizes the details of different processes.

A JEOL-5410 scanning electron microscope (SEM) was used to analyze the microstructure and compositions. X-ray mapping was done by high resolution hyper electron-probe microanalysis (FE-EPMA, JEOL JXA-8500F, Tokyo, Japan) to gain the distribution of elements of test sample. X-ray diffraction (XRD) patterns were obtained by a Rigaku ME 510-FM2 diffractometer operated at 30 kV and 20 mA. The range of scanning angle (2θ) was from 20° to 100°



Fig. 1. XRD analyses of Al_x CrFe_{1.5}MnNi_{0.5} alloys in the as-cast, as-homogenized, and as-rolled states: (a) x = 0.3 and (b) x = 0.5.

and the scanning rate was 4° /min. Vickers hardness measurement was conducted with a Matsuzawa Seiki MV-1 tester under a loading of 5 kg and a duration time of 15 s. Seven measurements were made on each sample for an average. Hot Vickers hardness measurement was performed with an Akashi AVK-HF hardness tester at RT, 300 °C, 500 °C, 700 °C, and 800 °C, respectively.

Oxidation resistance was obtained by isothermal oxidation treatment at 800 °C for different time intervals. Each datum point of weight gain was obtained by weighing the oxidized specimens after the treatment. Wear resistance was measured by the pin-on-belt wear test [4,6]. The belt was made of the Al_2O_3 emery cloth with grit #100. The testing was performed at a speed of 50 cm/s, a wear distance of 20 m, and a load of 5 kg. The wear resistance was calculated using the formula: $1/[(the mass of loss/density) \times 1000/wear distance], and has a unit of m/mm³.$

3. Results and discussion

3.1. X-ray diffraction analysis and hardness comparison

Fig. 1 shows the XRD patterns of $Al_xCrFe_{1.5}MnNi_{0.5}$ alloy in the as-cast, as-homogenized, and as-rolled states, respectively. The crystal structures of the $Al_{0.3}CrFe_{1.5}MnNi_{0.5}$ alloys in all states are

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