



## Effect of W and Zr on structural, thermal and magnetic properties of AlCoCrCuFeNi high entropy alloy

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### ABSTRACT

The equiatomic multicomponent AlCoCrCuFeNi hexanery high entropy alloy (HEA) was synthesized by mechanical alloying. The effect of the addition of W and Zr was studied by making equiatomic AlCoCrCuFeNiW septenary and AlCoCrCuFeNiWZr octonary high entropy alloys. The effect of alloying addition with milling time was studied using XRD, DSC/TGA, SEM, TEM and Mossbauer spectroscopy. Supersaturated solid solution structure was achieved after 60 h milling for hexanery alloy and after 40 h milling for both septenary and octonary high entropy alloys. The variation of Mossbauer parameters for AlCoCrCuFeNi system with the addition of W and Zr makes its magnetic environment more complex which is indicated by the conversion of magnetic AlCoCrCuFeNi HEA (60 h milling) to partially paramagnetic AlCoCrCuFeNiW (40 h milling) to completely paramagnetic AlCoCrCuFeNiWZr high entropy alloy.

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### 1. Introduction

The traditional concepts for alloy design are mainly based on one principal element to serve as a matrix for primary properties, with the addition of other minor elements to modify microstructure and properties e.g., steels, TiAl intermetallics, bulk metallic glasses (BMGs) [1,2]. The main reason for not incorporating multi-principal elements into alloy was the formation of many intermetallic compounds and complex structures which result in brittleness, difficulty in processing and complication in analysis [3–5].

In recent years, a new kind of alloys, high entropy alloys (HEAs) with multiple principal elements in equimolar or near-equimolar ratios, has been developed by Yeh et al. [4–7]. HEAs comprise of five or more principal elements and possess large mixing entropies ( $\Delta S_{\text{config}} \geq 1.61R$ ). The concentration of each principal element can vary between 5 and 35 at.%, with or without the addition of minor alloying elements (<5%). There can be countless combinations for HEAs in contrast to conventional alloy systems which are based on one principal element. Studies have shown that HEAs possess high mixing entropies, lattice distortion, sluggish diffusion and cocktail effects, which provide them with a wide spectrum of microstructure and properties depending on the composition and processing route [5,7]. Compared with traditional alloys, the high mixing entropy of HEAs greatly reduces the free energy of simple

solid solution phases and make them more stable than intermetallic compounds and other ordered phases [4–9]. HEAs show the formation of simple BCC and/or FCC solid solution structure with nanocrystalline and even amorphous phases [6–8,10,11]. The extraordinary physical and chemical properties of HEAs i.e. high hardness, very good ductility in as-cast state, excellent resistance to anneal softening, high strength up to 800 °C with enhanced plasticity, and good wear, corrosion and oxidation resistance, make them potential candidate for various industrial applications [5,7,12–15]. In addition, the coating technology would further expand the applications of HEAs as functional films, for example hard-facing for golf club heads and rollers, diffusion barriers and soft magnetic films [7,16,17]. Simple solid solution structure with very good properties and the new potential applications of HEAs have attracted the attention of many researchers. In order to understand the behavior of HEAs in a better way, new compositions are being developed by employing different synthesis routes and using more sensitive tools for their characterization. In the present study, equiatomic hexanery AlCoCrCuFeNi HEA was synthesized by mechanical alloying (MA). Effect on the structural, thermal and magnetic behavior of hexanery AlCoCrCuFeNi HEA was explored by the addition of tungsten and zirconium in this system.

### 2. Experimental

Elemental powders of Al, Co, Cr, Cu, Fe, Ni, W, Zr with purity higher than 99.5% and particle size of  $\leq 45 \mu\text{m}$  were used as starting materials. Three HEA compositions i.e. hexanery AlCoCrCuFeNi, septenary AlCoCrCuFeNiW and octonary AlCoCrCuFeNiWZr were prepared by mixing elemental powders in equiatomic ratios.

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The powders were milled for 60 h in high energy ball mill (NQM 20 Planetary Ball Mill) at 250 rpm. Stainless steel vials and balls with 10 mm diameter were used as milling media with ball-to-powder weight ratio of 15:1. In order to avoid excessive cold welding, n-heptane was used as process controlling agent (PCA) which also helps avoiding the alloys oxidation. To control the milling progress and confirm the alloy formation during MA, powder samples were collected in every 5 h intervals. Mechanically milled samples were analyzed using Bruker D8 Discover HR X-ray diffractometer (XRD) with Cu K $\alpha$ 1 radiation ( $\lambda = 1.5418 \text{ \AA}$ ) for investigating phase formation, crystallite size and lattice strain. The microstructure of the alloy powders was examined using scanning electron microscopy (SEM) attached with energy dispersive X-ray spectrometer (EDX) and transmission electron microscopy (TEM). In order to evaluate the effect of W and Zr addition on thermal characteristics of hexanery AlCoCrCuFeNi HEA, thermal analysis of all three HEAs was carried out from room temperature to 1400 °C using TA Instruments/SDT-Q600 differential scanning calorimeter (DSC) and thermal gravimetric analyzer (TGA) at heating rate of 20 K/min. Finally, Room temperature Mossbauer effect measurements were carried out using a  $^{57}\text{Co}$  (Rh-matrix) radiation source in transmission geometry. The data analysis was performed using a computer program MOS-90 [18], assuming that the peaks were Lorentzian in shape. The quality of data fitting was checked by the  $\chi^2$  - test.

### 3. Results and discussions

#### 3.1. XRD analysis

Fig. 1 shows the XRD patterns of the equiatomic AlCoCrCuFeNi (16.67 at.% each) hexanery high entropy alloy powders prepared under different milling durations. The primitive blending powder (0 h milling) includes diffraction peaks of all constituent elements. As the alloy is milled for 10 h, most of the elemental peaks disappear except the strongest ones. The disappearance of diffraction peaks at 10 h of milling can be considered as the beginning of the solid solution formation reaction which completes at 60 h of milling. The peak associated with the formation of solid solution depicts decreased intensity and broadening with milling time. This shows that as the milling time is increased, the alloy behavior continuously changes i.e. polycrystalline  $\rightarrow$  nanocrystalline  $\rightarrow$  amorphous. During MA process, the disappearance of peaks, intensity decrement and peak broadening can be attributed to refined crystallite size, accumulated lattice strains and decreased crystallinity [11,19,20]. Figs. 2 and 3 show the XRD patterns of the equiatomic septenary AlCoCrCuFeNiW (14.29 at.% each) and octonary AlCoCrCuFeNiWZr (12.5 at.% each) HEAs respectively. Marked changes in the XRD patterns can be observed with the introduction of W to hexanery AlCoCrCuFeNi alloy. MA for 10 h causes many elemental peaks to disappear except W while no further peak

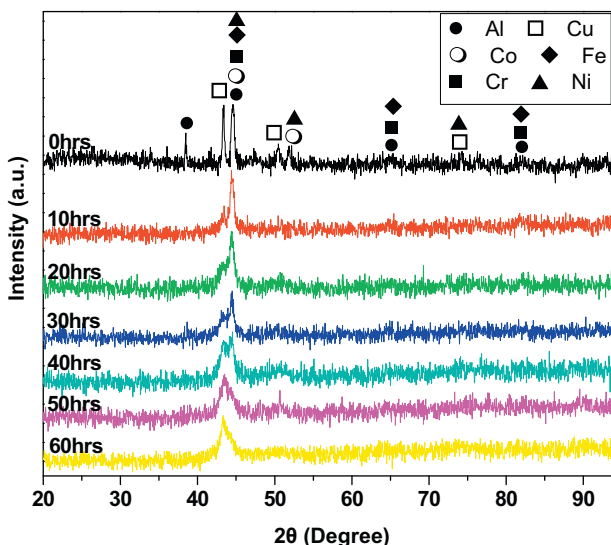


Fig. 1. XRD patterns of equiatomic AlCoCrCuFeNi HEA with varying milling time.

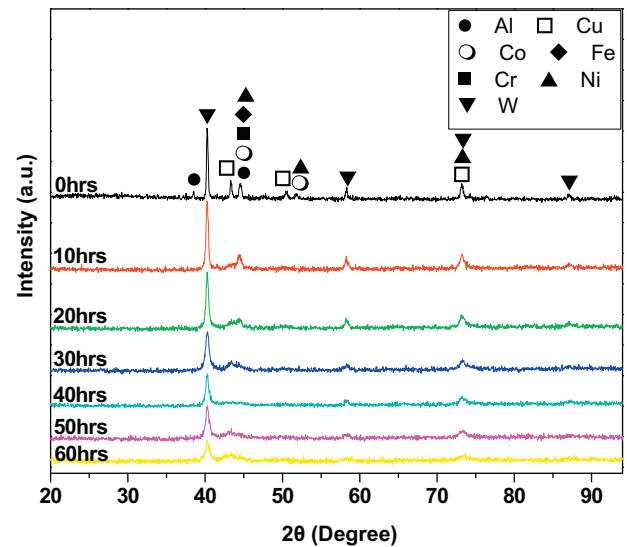


Fig. 2. XRD patterns of equiatomic AlCoCrCuFeNiW HEA with varying milling time.

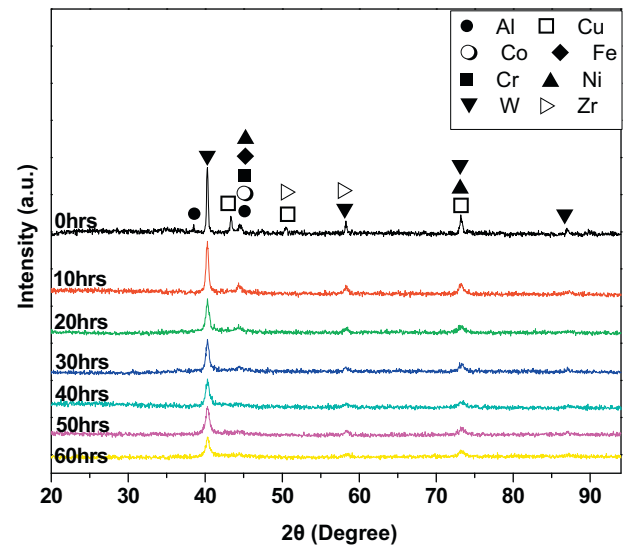


Fig. 3. XRD patterns of equiatomic AlCoCrCuFeNiWZr HEA with varying milling time.

disappearance was observed up to 30 h. Simple solid solution structure was achieved after 40 h of milling and remained unchanged up to 60 h of milling. On the other hand, octonary alloy, containing both W and Zr showed similar structural evolution behavior that was observed in case of septenary alloy. The complete solid solution structure was achieved after performing milling for 40 h. The different constituent elements of HEAs with different crystal structure and atomic size, as listed in Table 1 [21], compromises with each other to form the same BCC and/or FCC structure with no matrix or host element. All the atoms are regarded as solutes, thus expected to be randomly distributed in the crystal lattice [20]. Thermal properties of each element in the alloys are also shown in Table 1 [22], which gives an idea about the entropy of fusion of the elements in comparison with the enthalpy of mixing. This table clearly demonstrates that entropy of fusion of the elements present in the systems (maximum value of 11.5 J/mol K) is less than the configurational entropy of multi-component HEA systems (14.9 J/mol K for hexanery, 16.2 J/mol K for septenary and 17.3 J/mol K for octonary alloy), that favors the

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