Intermetallics 102 (2018) 58–64

Contents lists available at ScienceDirect

Intermetallics

journal homepage: www.elsevier.com/locate/intermet

Strengthening mechanism in a high-strength carbon-containing powder metallurgical high entropy alloy

Jiawen Wang[a], Bin Liu[a], C.T. Liu[b], Yong Liu[a,*]

[a] State Key Laboratory of Powder Metallurgy, Central South University, Changsha, 410083, PR China
[b] Department of Materials Science and Engineering, City University of Hong Kong, PR China

ARTICLE INFO

Keywords:
High entropy alloys
Strengthening
Microstructure
Mechanical properties
Powder metallurgy

ABSTRACT

A carbon-containing $FeCoCrNiAl_{0.5}$ high entropy alloys (HEAs) with high tensile strength was fabricated by powder metallurgy (P/M) method. The P/M process includes gas atomization and hot extrusion of pre-alloyed HEA powder. The microstructural evolution and mechanical properties were systematically investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and tensile tests. The results showed that the gas-atomized HEA powder was of dual phase, including face centered cubic (fcc) phase and B2 phase. Hot extrusion caused the precipitation of $M_{23}C_6$ carbides, the formation of dislocations and the refinement of microstructure. The as-extruded HEA exhibited a tensile strength as high as 1093 MPa and an elongation of ~12.4%. The contributions of different strengthening mechanisms were quantitatively calculated, and it was found that the grain boundary strengthening and the dislocation strengthening are the main strengthening mechanism.

1. Introduction

Developing high strength and low cost materials is a constant goal for material scientists. Recently, a new kind of alloys referred as high entropy alloys (HEAs) have received considerable attentions due to their unique structures and excellent mechanical properties [1–4]. Originally, HEAs were designed to mix multiple elements in equiatomic or near-equiatomic ratios, and to form a stable single-phase structure through the effect of the high configurational entropy [4–6]. A typical single-phase HEA CrMnFeCoNi exhibits exceptionally high damage tolerance and fracture toughness even at cryogenic temperatures [7]. Nevertheless, a number of studies show that a single-phase matrix only with intrinsic characteristics of HEAs is relatively weak in strength for practical applications [8]. Motivated by these findings, various strengthening mechanisms have been introduced by thermo-mechanical treatments to enhance the strength of HEAs. For example, the increment of yield strength about 100 MPa was obtained in an $Al_{0.3}CrFeCoNi$ HEA by grain boundary strengthening [9]. The yield strength of a CrMnFeCoNi HEA increases from 170 MPa to 360 MPa, when the grain size decreases from 155 μm to 4 μm [10]. Nanocrystalline $Al_{7.5}Fe_{25}Co_{25}Ni_{25}Cu_{17.5}$ HEA produced by mechanical alloying (MA) and spark plasma sintering (SPS) exhibits a very high compressive strength [11]. In addition to the grain boundary strengthening, the precipitation strengthening is another way to strengthen HEAs. For example, homogeneous $L1_2$ Ni_3(Ti, Al)-type precipitates in the $(FeCoNiCr)_{94}Ti_2Al_4$ HEA contribute a strength increment of about 320 MPa [12]. The precipitations of hard σ and μ intermetallic phases were introduced in $CoCrFeNiMo_x$ HEAs by thermo-mechanical treatments, producing a strength of 1186 MPa and an elongation of 18.9% [13]. The strength and the ductility of the dual-phase $Fe_{80-x}Mn_xCo_{10}Cr_{10}$ HEAs both increase with the decrease of the grain size of face centered cubic (fcc) phase and the increased fraction of hcp phase [14]. Wang et al. [15] made attempts to improve the strength of 1.1 at. % carbon-doped $Fe_{40.4}Ni_{11.3}Mn_{34.8}Al_{7.5}Cr_6$ HEAs by multiple strengthening mechanisms, and found that the reduction in the grain size resulted in a sharp increase in strength, while the precipitation, produced only a slight increase in strength and a decrease in ductility. So far, simultaneous additions of carbon and strong carbide-forming elements, for example, Mo, Nb and Ta, are quite few, which can expect to induce the combination of solid solution strengthening and other strengthening effects.

Casting technique is the most widely adopted route for the synthesis of HEAs [16–19]; however, the coarse and heterogeneous microstructures need to be improved [19–22]. Therefore, an additional thermo-mechanical process becomes a necessary step to optimize the microstructures and enhance the mechanical properties. Besides, the complex alloying composition and the addition of carbon are also a great challenge for casting HEAs. Powder metallurgy (P/M) is recently reported to be a promising way for preparing HEAs [23,24]. In this

* Corresponding author.
E-mail address: yonliu@csu.edu.cn (Y. Liu).

https://doi.org/10.1016/j.intermet.2018.07.016
Received 15 March 2018; Received in revised form 20 June 2018; Accepted 26 July 2018

work, a high performance HEA was prepared by hot extruding pre-alloyed powder, and the microstructural evolution and strengthening mechanisms were investigated to achieve in-depth understanding of HEA.

2. Experimental

2.1. Fabrication of pre-alloyed powder

High-purity elemental metals of a nominal composition-$Al_{10.5}Cr_{21.1}Fe_{21.1}Co_{21.1}Ni_{21.1}Mo_{2.5}C_{2.5}$ (at.%) were melted in a vacuum induction furnace. The melt then dropped through a ceramic tube, and was atomized by high pressure argon. The atomization pressure was 4 MPa. The gas-atomized powder was kept in an airtight chamber until cooling down. The composition of powder was analyzed by chemical methods. The oxygen content was determined by the fusion method on a Leco O/N analyzer.

2.2. Hot extrusion

The gas-atomized HEA powder was filled into a stainless steel can with dimensions of *d* 60 mm × 150 mm. The can was degassed for 12 h at 500 °C and sealed in vacuum. The encapsulated powder was subsequently pre-heated at 1150 °C for 1 h, and immediately hot extruded into bars with an extrusion ratio of 6:1. After hot extrusion, the bars were cooled in air.

2.3. Microstructural characterization

Specimens for analyses were sectioned along the extrusion direction (ED). X-ray diffraction (XRD) tests were performed on a Rigaku D/max 2550VB diffractometer using Cu-Ka radiation. An FEI Quanta 250 field emission gun (FEG) scanning electron microscopy (SEM) with backscattered electron (BSE) mode was employed to examine the microstructures of the as-extruded HEA. Specimens for SEM examination were ground using silicon carbide papers, and then polished using a 0.05 μm colloidal silica suspension. Electron backscatter scattered diffraction (EBSD) measurements were carried out by an FEI Quanta 650 FEG SEM equipped with a fully automatic HKL technology EBSD attachment. Data acquisition and post-processing were performed using both the Aztec and Channel 5 software. For EBSD specimens, vibration polishing was applied for about 4 h after standard metallographic procedure. The foils for transmission electron microscopy (TEM) analyses were thinned using Struers Tenupol 5 in an electrolyte of 25% nitric acid and 75% methanol with a voltage of 10 V at −25 °C. TEM investigations were conducted on electrochemically polished disks using a FEI Tecnai F20 TEM operated at 200 kV.

2.4. Mechanical property tests

Cylinder specimens for tensile tests, with a gauge size of *d* 4 mm × 20 mm, were machined from the as-extruded HEA bars along the ED. All specimens were mechanically polished prior to tensile tests in order to remove surface irregularities and to guarantee an accurate determination of the cross-sectional area. The quasi-static tensile tests were performed on an MTS landmark testing machine with a strain rate of 1×10^{-3} s^{-1}.

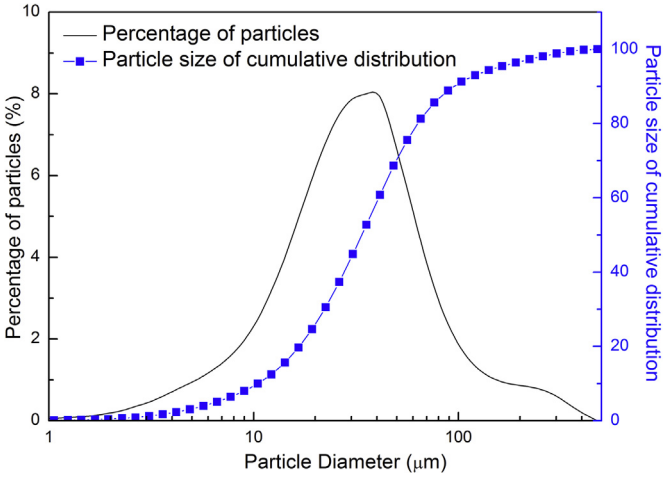

Fig. 1. Particle size distribution of gas-atomized HEA powder.

3. Results

3.1. Characterization of HEA powder

The chemical analyses of the gas-atomized HEA powder (see Table 1) suggest that the real composition is in good agreement with the nominal composition. Additionally, the oxygen content of the powder is as low as 175 ppm, indicating no obvious oxidation during the gas atomization. As shown in Fig. 1, the mean particle diameter (*d*50) of the gas-atomized HEA powder is about 33.73 μm, with a distribution from several microns to 140 μm. Fig. 2(a) shows the morphology of the gas-atomized HEA powder. It can be seen that the powder is predominantly spherical or near-spherical in shape with some satellite particles. The surface and the internal microstructures of the gas-atomized HEA powder are both dendritic in submicron scale, as shown in Fig. 2(b) and (c), respectively. The X-ray diffraction (XRD) patterns in Fig. 3 reveal the phase constitution of the gas-atomized HEA powder. It is clear that the powder consists of fcc phase and B2 phase.

3.2. Microstructures of the as-extruded HEA

Fig. 4 shows the XRD patterns and SEM images of the longitudinal section of the as-extruded HEA bars. After hot extrusion, some peaks associated with $M_{23}C_6$ carbide appear in the XRD patterns. The phase composition of the as-extruded HEA consists of fcc phase, B2 phase and $M_{23}C_6$ carbide (see Fig. 4(a)). Meanwhile, the abnormal diffraction peak intensity of fcc phase suggests that the texture along [200] was formed. The gas-atomized HEA powder exhibits a good flowability and deformability during the hot extrusion. The particles were consolidated to nearly full density, and only very few pores can be observed in the microstructure (see Fig. 4(b)). Combined with the XRD patterns, it can be concluded that the main phase is fcc, the grey roughly circular phase is $M_{23}C_6$ carbides, and the dark phase is B2. The high magnification image and corresponding compositional profiles displayed in Fig. 4(c) and Table 2 show that the $M_{23}C_6$ carbide contains high amount of Cr and Mo, and the B2 phase is enriched in Ni and Al. The distribution of the elements in fcc matrix is homogeneous. Fig. 5(a) through (c) are EBSD inverse pole figure (IPF) map, phase color (PC) map and kernel

Table 1
Chemical compositions of gas-atomized HEA powder.

Element	Al	Cr	Fe	Co	Ni	Mo	C	O
Nominal composition (at.%)	10.5	21.1	21.1	21.1	21.1	2.5	2.5	–
Measured composition (at.%)	10.42	21.38	23.49	19.69	19.89	2.51	2.62	0.0175