Intermetallics 75 (2016) 25–30

Contents lists available at ScienceDirect

Intermetallics

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

Microstructure and mechanical properties of equimolar FeCoCrNi high entropy alloy prepared via powder extrusion

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ARTICLE INFO

Article history:
Received 19 February 2016
Received in revised form
23 April 2016
Accepted 21 May 2016
Available online 1 June 2016

Keywords:
High entropy alloys
Powder metallurgy
Extrusion
Microstructures
Mechanical properties
Electron backscatter diffraction

ABSTRACT

An equiatomic FeCoCrNi high alloy (HEA) with both high tensile strength and ductility was produced by a powder metallurgy (P/M) method. The P/M process includes a gas atomization and a hot extrusion of pre-alloyed HEA powder. Microstructures and mechanical properties were characterized using optical microscopy (OP), scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), and tensile tests. The results show that the P/M FeCoCrNi HEA has a single face centered cubic (fcc) structure and an equiaxed microstructure. No obvious porosity and brittle intermetallic phase was found. The as-extruded alloy exhibits a very high tensile strength of 712.5 MPa, and still maintains an elongation as high as 56%. The improvement of the tensile properties is caused by the solid solution strengthening, grain boundary strengthening and homogenous microstructure. Therefore, the powder hot extrusion can be considered as a promising way for preparing large-sized HEAs with high mechanical properties.

1. Introduction

High entropy alloys (HEA) consist of more than four elements mixed in near equiatomic proportions. Therefore, from the alloy designing perspective, HEAs are radically different because conventional alloys are always based on one dominant element [1–5]. With a high configurational entropy in the solid solution state, the HEAs can have phase structures of face centered cubic (fcc) and body centered cubic (bcc), as well as some secondary phases, such as hexagonal close packed (hcp) solid solutions, nano sized precipitates, and even amorphous phases [6–9]. The distinctive structure and phase composition provide HEAs with multiple excellent properties, such as high hardness/strength, excellent corrosion and oxidation resistance, high thermal stability, superior fatigue resistance, excellent magnetic properties and exceptional high temperature strength, as well as enhanced hydrogen storage [1,4,10–13].

Mostly, HEAs are processed by the casting route, however, since the alloys contain multiple elements, the compositional segregation of high melting point elements and the evaporation of low melting point elements are hard to avoid during the melting process [4,8]. Moreover, HEAs ingots have coarse dendritic structures with the precipitations of some brittle intermetallic phases [4]. These intrinsic processing problems are of great harm to the mechanical properties and the industrial application of HEAs. Powder metallurgy (P/M), which is considered as a low cost and high efficiency technique, is widely employed for the synthesis of non-equilibrium materials. Recent works [14,15] show that the HEAs can be made by mechanical alloying (MA) and subsequent consolidation process, such as spark plasma sintering (SPS), and hot pressing. However, since HEAs contain high content of alloying elements, the alloying and homogenization processes take a long time of milling, for example 20–60 h [16,17]. Moreover, in the whole milling process, the powder can easily be contaminated by the milling medium and the environment, and thus, the mechanical properties, especially the ductility, can be deteriorated. Gas atomization is a widely used way for making metallic powder by the rapid solidification process, in which the cooling rate can be in the range of 10^5–10^6 °C/s [18]. The rapid cooling rate helps to prevent the compositional segregation, to refine the microstructure and to stabilize the single phase or the amorphous phase. Compared with

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http://dx.doi.org/10.1016/j.intermet.2016.05.006
0966-9795/

mechanically alloyed powder, gas-atomized powder is of higher purity and more homogeneous both in the composition and the morphology. Therefore, gas-atomized HEA powder is a good candidate for making bulk HEA materials. The gas-atomized HEA powder actually is hard for densification due to its high strength and spherical morphology. Hot extrusion can be used for densification of the hard-to-sinter powders. For example, Kawamura et al. [19] prepared $Zr_{65}Al_{10}Ni_{10}Cu_{10}$ (at.%) glassy alloy by powder extrusion with gas-atomized powders and found that the relative density of the extruded alloy can reach to 99.2%. Liang et al. [20] prepared Ti6Al4V bars via powder compact extrusion with gas-atomized powders and found that the relative density, ultimate tensile strength (1300 MPa), and elongation (10%) are all at a high level. So far, no such work was performed on gas-atomized HEA powder.

For the mechanical properties of P/M HEAs, most studies were focused on the compression tests or hardness [4,14,21,22] because the as-prepared P/M billets were usually not large enough for making tensile samples. In our previous work [14], we studied the tensile properties of the P/M FeCoCrNiMn alloy prepared by MA and SPS, and found that the P/M HEA have a tensile strength as high as 1050 MPa at ambient temperature, however, it has limited tensile ductility due to the high impurity contents.

In this work, FeCoCrNi HEA powders were prepared by gas atomization, and then, powder hot extrusion was performed in order to obtain a high density and a low impurity content. The microstructural evolutions and the mechanical properties of the P/M FeCoCrNi HEA were investigated.

2. Experimental

2.1. Fabrication of pre-alloyed powders

High purity Fe, Co, Cr and Ni of an equiatomic ratio were melted in an induction heated vacuum furnace. The melt was then drop through a ceramic tube, and atomized by high purity Ar. The atomization pressure was 4 MPa. The liquid droplets flied in the atomization chamber, cooled down and solidified to powders. The composition of the gas-atomized powder was analyzed by chemical methods. The oxygen content was determined by the fusion method on a Leco O/N analyzer.

2.2. Powder extrusion

The powder hot extrusion process is illustrated in Fig. 1. The as-prepared powder were filled into a stainless steel can with the dimensions of *d* 60 mm × 150 mm. The can was degassed at 500 °C for 12 h and sealed in vacuum. Then the encapsulated powders were pre-heated at 1473 K for 60 min, and immediately subjected to hot extrusion with an extrusion ratio of 9.5 and a velocity of ~10 mm/s on a 2500 T hydraulic press. After extrusion, the billets were cooled in air.

2.3. Microstructural characterization

Microstructure was observed using a Leica optical microscope (OM), a field emission scanning electron microscope (FESEM) (FEI Nova Nano230, USA). Crystallographic analysis was carried out by electron backscattered diffraction (EBSD) using an orientation imaging microscope attached to the FESEM. The acceleration voltage during the FESEM measurements was 30 kV, and the beam current was approximately 100 μA. Phase constitutions were identified by X-ray diffractometer (XRD) (Rigaku D/MAX-2250, Japan) with a Cu/Ka radiation. The sintered density was measured by Archimedes method, and the relative density was measured by the imaging processing of porosity in the cross section of the samples under the optical microscope.

2.4. Mechanical property tests

Tensile samples with a gage size of *d* 4 mm × 15 mm were cut from the extruded HEA bars along the extrusion direction (ED). Tensile tests were performed on an Instron 3369 testing machine with a loading strain rate of 10^{-3}/s. The engineering stress-strain curve was plotted according to the recorded data.

3. Results

3.1. Characterization of the powders

Table 1 shows the chemical analyses of the gas-atomized FeCoCrNi HEA powder. The results show that the actual composition is in good agreement with the nominal composition. The content of oxygen is as low as 720 ppm, indicating no obvious oxidation during the gas atomization. The mean particle size (*d*50) of the gas-atomized powder is about 35.2 μm, with a distribution from several microns to more than 100 μm, as shown in Fig. 2. Fig. 3 shows the morphology and microstructure of the gas-atomized powder. The powders are in spherical shape with some satellite structure. During the gas atomization, smaller melt droplets experienced a higher cooling rate and solidified in a shorter time. They can easily stick to the surface of large droplets which are still in a mushy state and formed the satellite structure. The magnified image in Fig. 3b shows that the powders consist of very fine cellular structure at submicron scale, which is formed due to the fast solidification process [23]. Fig. 4 shows the XRD pattern of the gas-atomized FeCoCrNi HEA powders. Only fcc phase peaks (lattice parameter = 3.59 Å) can be observed, indicating a single phase solid

Table 1
Chemical compositions of gas-atomized FeCoCrNi HEA powder.

Elements	Fe	Co	Cr	Ni
at.%	24.5	24.4	24.1	27

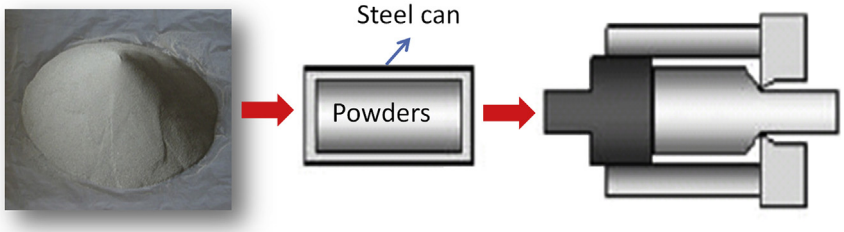

Fig. 1. Depiction of preparation process of P/M FeCoCrNi HEA.