Advanced Powder Technology 27 (2016) 1128–1133

Contents lists available at ScienceDirect

Advanced Powder Technology

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

Original Research Paper

Electromagnetic wave absorption properties of mechanically alloyed FeCoNiCrAl high entropy alloy powders

Peipei Yang, Ying Liu *, Xiuchen Zhao, Jingwei Cheng, Hong Li

School of Materials Science and Engineering, Beijing Institute of Technology, PR China

ARTICLE INFO

Article history:
Received 16 June 2015
Received in revised form 14 March 2016
Accepted 26 March 2016
Available online 4 April 2016

Keywords:
FeCoNiCrAl high entropy alloy powders
Mechanical alloying
Electromagnetic wave absorption properties
Reflection loss

ABSTRACT

FeCoNiCrAl high entropy alloy powders with flaky shape were prepared by mechanical alloying in a planetary ball milling machine for 70 h. The electromagnetic response behavior and electromagnetic wave absorption property of the powders (70 wt% powders) dispersed in the paraffin matrix were investigated as a function of frequency in the range of 2–18 GHz. We found that the calculated reflection loss (RL) was shown that the minimum RL reached −35.3 dB at 10.35 GHz with a matching layer thickness of 1.5 mm and an effective absorption bandwidth of 2.7 GHz (9.2–11.9 GHz), indicating FeCoNiCrAl high entropy alloy powders have excellent electromagnetic wave absorption properties.

1. Introduction

A high entropy alloy (HEA) is originally defined as an alloy system, composed of five or more kinds of principal elements in an equimolar or near equimolar ratio, which the elements have a small difference in atom radii (<15%) and concentration varying from 5 to 35 at.% [1]. The high entropy of mixing of HEAs can stabilize disordered solid solution phases with simple structures, such as body-centered cubic (BCC) or/and a face-centered cubic (FCC), in comparison with ordered crystalline intermetallic phases which often contain giant and structurally complex unit cells [2–4]. With proper composition designing, the HEAs exhibit high hardness, excellent ductility as well as promising resistances to wear, oxidation and corrosion. As a promising HEA, FeCoNiCrAl system was usually investigated for its relationship between the microstructure and properties [5–7]. Fu et al. reported the preparation of FeCoNiCrAl HEA and the effect of heat treatment on its Vickers hardness and compressive strength [8]. Meanwhile, magnetic property of the FeCoNiCrAl has also been investigated, for example, Kao investigated the electrical and magnetic properties of $FeCoNiCrAl_x$ HEAs [9]. The HEAs powders have been synthesized successfully by MA, and the ball milling parameters affect the microstructure and even the properties. With increasing the milling time, the amorphous alloy powders were fabricated, which had attractive soft magnetic properties [10–12]. However, to our best knowledge, the electromagnetic (EM) wave absorption properties of FeCoNiCrAl HEA have not been reported yet.

Recently, some new EM wave absorption materials have been investigated [13–15], Zhu et al. reported the preparation of graphene oxide–nickel composites and the minimum reflection loss was −42 dB at 17.6 GHz with a thickness of 2 mm [16]. Zhao and coworkers synthesized one-dimension Ni chains by a facile solvothermal method and the minimum reflection loss was −19.9 dB with a thickness of 0.8 mm at 17.2 GHz [17]. However, their practical applications have been somewhat limited due to their complex formation process, low yield or high cost. Compared to these materials, high entropy alloys have unbeatable advantages in this application field.

In our previous work, several kinds of morphologies and crystal structures of cobalt particles were investigated as EM wave absorption materials. We found that the ferromagnetic materials with high symmetrical crystal structure, laminated structure, or high special surface area have excellent EM wave absorption properties [18–20]. In the present paper, we report the synthesis of FeCoNiCrAl HEA powders through mechanical alloying (MA). The EM wave absorption property of FeCoNiCrAl HEA powders are investigated as a function of frequency from 2 to 18 GHz. The calculated reflection loss (RL) indicates the powders have a potential application for EM wave absorption.

2. Experimental method

FeCoNiCrAl HEA powders were prepared through mechanical alloying (MA) in the anhydrous ethanol acting as process control

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

http://dx.doi.org/10.1016/j.apt.2016.03.023
0921-8831/

agent. The elemental powders (Fe, Co, Ni, Cr, and Al) are in equimolar ratio with particle size of 1–5 μm and purity of 99.5 wt%. MA was performed in a planetary ball milling machine (QM-3SP4L; Nanjing Nanda Instrument Plant) for 70 h at a speed of 250 rpm. The effective volume of container was 500 mL and the ball to powder weight ratio (BPR) was 15:1 with initial powder of 20 g. The loaded zirconium dioxide balls were in four sizes (i.e. φ = 15 mm, mass = 30 g; φ = 12 mm, mass = 60 g; φ = 10 mm, mass = 105 g and φ = 8 mm, mass = 105 g).

For phase analysis, XRD analysis was carried out on FeCoNiCrAl HEA powders with milling time up to 70 h at an interval of 10 h using Rigaku Ultima-III X-ray diffractometer with Cu Kα radiation (λ = 1.5406 Å) over 20–90°. The morphology of powders was performed by scanning electron microscopy (SEM, QUANTA FEG 250) with EDS. The magnetic properties were determined by a vibrating sample magnetometer (VSM) at room temperature. The EM response behaviors of FeCoNiCrAl HEA powders, including complex permittivity ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and complex permeability ($\mu_r = \mu' - j\mu''$), were determined by an HP 8722ESS vector network analyzer using the T/R coaxial line method with frequency range of 2–18 GHz according to the Nicolson and Ross and Weir and Baker-Jarvis et al's theory. The samples were a ring shaped (i.d. = 3 mm and o.d. = 7 mm, thickness = 2 mm) with powders (70 wt%) uniformly dispersing in paraffin matrix.

3. Results and discussion

The crystal structure of as-synthesized powders with various milling time is determined by XRD as given in Fig. 1a. Diffraction patterns of all the pure elements are predominantly observed in the initiate time (0 h). The peak of Al at 78° disappears when the milling time extends to 10 h, and the peaks of Al at 38° and Co at 42° disappear simultaneously with drastic decrement of diffraction intensity with 30 h-milling. The disappearance of diffraction peaks of Al and Co were considered as the beginning of the solid solution formation. After 50 h milling, the peaks are almost the same as those of 30 h-milled powders except for a minor decrement in intensity. The peak of Co at 47.5° disappears as the milling time extends to 60 h. Extending ball milling time from 60 h to 70 h, the microstructure of the powders is little changed. The powders are just polycrystalline mixtures as indicated by the sharp Bragg-peaks corresponding to BCC ((110), (200), (211)) and FCC ((111), (200), (220)) structures and a detailed analysis of peak positions (2θ = 44–45°) given in Fig. 1b also confirms the main diffraction peak pattern corresponding to FCC and BCC phase, indicating the formation of complete solid solution (high entropy alloy) [21–24]. It is possibly explained that the elements of Al and Co possess lower melting point compared with other elements, which lead to dissolve rapidly. Meanwhile, Fe and Cr are in BCC crystal structures and Ni is FCC structure leading to accommodate other elements with the same crystal structure or the similar atomic radius. Therefore, Co appears to dissolve into Fe and Cr and Al dissolves into Ni as the milling time increases during MA process. Moreover, Cr has the highest melting point compared with other elements leading to dissolve most slowly [25,26]. The crystallite size after different milling time has been calculated by Scherrer's formula. As shown in Table 1, the crystallite size is refined as the milling duration increases and reaches 26.68 nm after 70 h milling. The crystallite size of the powders is almost the same for 60 h-milled and 70 h-milled powders indicating that the stable of the MA process has been achieved. Throughout the milling process, the intensity decreasing, peak broadening and its subsequent disappearance may result from refined crystal size, high lattice strain and changed crystallinity [8]. As a result, the density of the defects, such as dislocations, stacking-faults and grain boundaries, increases during the MA process, which is beneficial to improve the EM properties [27].

The morphological evolution of FeCoNiCrAl HEA powders under different milling time is shown in Fig. 2. It could be seen that the powders shape changes significantly with the increasing milling time. The raw powders are in nearly spherical shape with a granular size of 1–5 μm (Fig. 2a). In the early period of MA (Fig. 2b), the powders are cold welded together to form larger powders than that of raw powders and changed into flaky or platelet shape partly, which lead to the aspect ratio increase and possibly change the EM parameter simultaneously [28]. Subsequently, with increasing milling time (Fig. 2c and d), most of the cold welded agglomerations with flaky shape are crushed down to smaller size and the thicknesses of powders also decrease. After 70 h milling, the mechanical alloying powders reveal an average particle size of approximately 1–5 μm and flake thickness of 0.2–1 μm as shown in Fig. 2e. The circulation of crushing and cold welding gradually reduces the crystalline size, and facilitates the diffusion and alloying among different elements [29]. The corresponding EDS spectrum of prepared FeCoNiCrAl HEA powders of 70 h-milling is illustrated in Fig. 3. Only signals of Fe, Co, Ni, Cr and Al can be observed. In the mixture of raw materials, the nominal composition of each element is nearly 20%, and the chemical composition of final powders is relatively similar to the initial powder, indicating that the contamination level is negligible. Combining results of XRD and EDS, the FeCoNiCrAl HEA powders are successfully prepared by MA.

The magnetic property of as-milled powders after 70 h-milling is recorded at room temperature by VSM with an applied field of −10 kOe to 10 kOe. Fig. 4 shows the magnetic hysteresis loop of the powders. It presents a ferromagnetic behavior. It has been shown that saturation magnetization (M_s) and coercivity (H_c) are

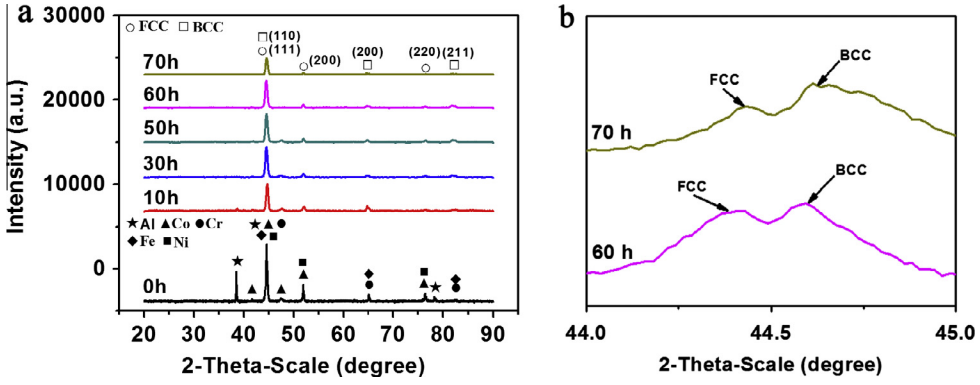

Fig. 1. (a) XRD pattern of FeCoNiCrAl HEA powders with different milling time and (b) the detailed scans for FeCoNiCrAl HEA powders in the range of 2θ = 44–45°.