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Bulk amorphous and nanocrystalline $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ alloy by rapid consolidation at super-high pressure

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Abstract

Microstructure and soft magnetic properties of a bulk amorphous and nanocrystalline $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ alloy prepared by rapid consolidation at a super-high pressure (at 1150 W at 5.5 GPa for 3 min) were investigated. The bulk alloy had a high relative density of 98.3% and high saturation magnetization of 120 emu/g. The size of the nanocrystalline α-Fe phase in the bulk samples was 10–16 nm. The relative density ρ_r had a linear relation with the crystalline volume fraction V_c in the bulk alloy: $\rho_r = 0.6624 + 0.5616V_c$. The preparing process used in this study makes it easy not only to generate the bulk alloy containing both amorphous and nanocrystalline phases but also to give good soft magnetic properties. And what is more important, there will be no longer restriction in the composition, size and shape of bulk alloys prepared by this process.

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Keywords: Bulk amorphous and nanocrystalline alloy; Super-high-pressure consolidation; Magnetic property

1. Introduction

Amorphous and nanocrystalline Fe-based alloys are technologically important materials due to their good magnetic properties and low cost [1–4]. However, the appearances of the alloys obtained by melt spinning and sputtering techniques are limited to ribbon and strip, film, wire, and powders, which largely restricts their applications in industries. Thus, generating bulk soft magnetic materials having amorphous and nanocrystalline microstructures is of great interest. Some research groups have reported the investigations with regard to the preparation of bulk soft magnetic

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amorphous materials [5–8]. But, to our knowledge, papers describing consolidation under the condition of super-high pressure by which bulk soft magnetic nanocrystalline alloys are prepared could not yet be found in the literature.

This paper presents the preparation, microstructure and magnetic property of a bulk amorphous and nanocrystalline $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ alloy fabricated by consolidation at a super-high pressure. The preparing process makes the features of simpler, more reliable, and particularly no restriction of compositions, size and shape of the alloys to be prepared.

2. Experimental

The alloy used in this paper was a newly developed $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ by the present authors, whose nanocrystallized ribbons showed a good combined soft magnetic property [9]. The amorphous ribbons were prepared by the melt spinning technique. The as-quenched ribbons were milled into powders using a planetary ball machine with a rotation speed of 450 rpm at −30–0 °C for 30 min and then consolidated into bulk samples (Φ20 mm × 10 mm) in a special multiaxial hydro-forming apparatus (HTDS-032A) operated with input current powers P_w of 530–1150 W and a pressure P of 5.5 GPa for a pressing time t of 3 min.

A differential scanning calorimeter (DSC) was used to test the crystallization temperature of the as-milled powders with a heating rate of 20 °C min. Density was measured by the Archimedean method, whose error was less than ±0.5%. Microstructures in the bulk samples were examined by means of a transmission electron microscope (TEM) and X-ray diffractometer (XRD) with Cu-K_α radiation. A vibrating sample magnetometer was employed to measure magnetic properties under an applied field of 15,000 Oe.

3. Results and discussion

3.1. DSC

Fig. 1 shows the DSC curve for the as-milled powders. Two exothermic peaks can be seen separately around 500 and 780 °C. The DSC curve was similar in shape to that of the as-quenched amorphous ribbons, except that the first exothermic peak was slightly broadened which is probably due to the relaxation of the stress and lattice distortion created during milling.

In the present study, consolidation temperature is very difficult to be accurately tested because of the multiaxial pressing hydro-forming apparatus. We cannot but make a suggestion that the more the input current power P_w, the higher the temperature in the being consolidated sample (as a reference, in another similar experiment for other alloys, the sample temperatures were estimated to be about 465±30, 530±30, and 585±30 °C when the applied P_w was 530, 820, and 1150 W, respectively). Based on the heating curve in Fig. 1 and pretests, the consolidating parameters used in this study were selected as follows: $P = 5.5$ GPa, $P_w = 530$–1150 W, $t = 3$ min.

3.2. XRD

The XRD patterns of the $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ bulk alloy prepared at different input current powers are represented in Fig. 2. The pattern of the as-milled powders is also shown for comparison. It is obvious that the as-milled powders consisted of

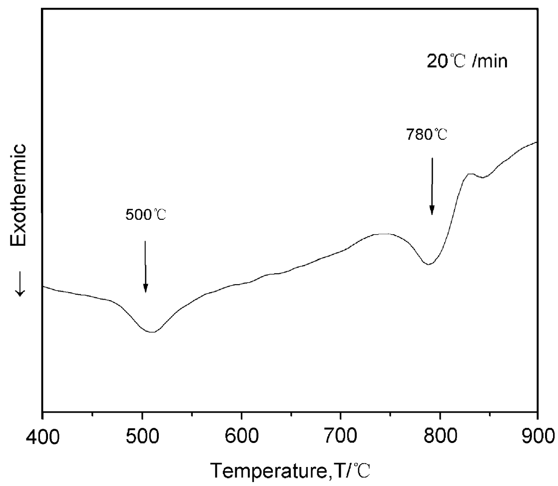

Fig. 1. DSC heating curve of the as-milled $Fe_{86}Zr_{5.5}Nb_{5.5}B_3$ powders.