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Review

Effects of magnetic field heat treatment on $Sm-Co/\alpha$ -Fe nanocomposite permanent magnetic materials prepared by high energy ball milling



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Yanfeng Su ^{a, b}, Hao Su ^b, Yuejin Zhu ^a, Fang Wang ^c, Juan Du ^b, Weixing Xia ^b, Aru Yan ^b, J.Ping Liu ^b, Jian Zhang ^{b, *}

^a Faculty of Science, Ningbo University, Ningbo 315211, China

^b Key Laboratory of Magnetic Materials and Devices, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Ningbo 315201, China

^c School of Materials Science and Engineering, NingBo University of Technology, Ningbo, Zhe Jiang Province, China

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ABSTRACT

Effects of magnetic field heat treatment on the structure and magnetic properties of Sm–Co/ α -Fe nanocomposite permanent magnetic materials fabricated by high energy ball milling are investigated in the present work. After a magnetic field heat treatment below 700 °C on as-milled amorphous Sm–Co/ α -Fe samples, the nanocomposite magnets with strong hard and soft magnetic interaction, showing a hysteresis loop of single phase characteristic, are obtained. The coercivity increases with the increase of annealing temperature. The coercivity, remanence and remanence ratio of the Sm–Co/Fe nanocomposite magnets are all enhanced after a heat treatment at a magnetic field as compared with those of nanocomposite magnets heat treated without a magnetic field. X ray diffraction analysis shows that the diffusion between the Sm–Co hard and α -Fe soft phases is suppressed by the magnetic field applied during the heat treatment process, leading to the inhibition of the grain growth of nanocrystal Sm–Co and α -Fe phases, and a finer nanostructure is obtained. Thus, a higher coercivity, remanence and remanence ratio are realized in Sm–Co/ α -Fe nanocomposite magnets after the magnetic field heat treatment also makes the direction of c axis of Sm–Co hard grains along the heat treatment magnetic field direction, leading to an enhancement of magnetic anisotropy of the Sm–Co/Fe nanocomposite magnets.

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1. Introduction

The strong exchange coupling at nanoscale between a hard phase with a high magnetocrystalline anisotropy field and a soft phase with a high saturation magnetization makes the possibility to achieve both high remanence and coercivity in the nanocomposite permanent magnetic material. The theoretical calculation predicts an extremely large maximum energy product (BH)_{max} of more than 100 MGOe for the nanocomposite permanent magnetic material which let it become a good candidate for the next generation of permanent magnetic material and attracted much attention in the last decades [1–5]. The nanocomposite permanent

* Corresponding author. E-mail address: zhangj@nimte.ac.cn (J. Zhang). magnetic materials are usually fabricated by the techniques such as mechanical alloying, rapid quenching and thin film deposition methods. The keys to obtain good magnetic performance are the hard magnetic phase orientation (anisotropic magnets) and an appropriate microstructure such as the grain sizes of hard and soft phases, interface and so on [4–8]. SmCo₅ has a very large magnetocrystalline anisotropy field and it is a good candidate for hard phase in nanocomposite magnets. High energy ball milling can make the nano sized α -Fe soft grains well distributed in the amorphous Sm–Co matrix [6]. After a heat treatment, the nanocomposite with nano sized Sm–Co and α -Fe phases are obtained [7]. However, how to further control the crystallized grain sizes of hard and soft phases to get a fine microstructure and how to prepare the anisotropic nanocomposite magnet with a textured hard grains are still a big challenge.

The magnetic heat treatment was used in the process of

fabricating magnetic materials [8], especially in the preparation of soft magnetic materials [9]. Atoms can be rearranged in a very small local area by the magnetic field blow the Curie temperature of the material, resulting in a preferred orientation along the direction of magnetic field and thus anisotropy structure is induced [10]. Ji et al. carried out a magnetic field heat treatment on the Nd₂Fe₁₄B/ α -Fe magnet near the Curie temperature of Nd₂Fe₁₄B [11]. They found that the magnetic field plays a certain role in the orientation of hard phase and can improve the microstructure of the $Nd_2Fe_{14}B/\alpha$ -Fe magnet. However, the crystallization is not complete due to the crystallization temperature of Nd₂Fe₁₄B much higher than its Curie temperature. Diffusion from α -Fe to the Nd₂Fe₁₄B phase also destructs its anisotropic structure. All these factors lead to a poor magnetic performance. The crystallization temperature of Sm-Co amorphous phase is about 500 °C [12], while the Curie temperature of SmCo₅ is about 720 °C. Due to the lower crystallization temperature than their Curie temperature for Sm-Co phases, the magnetic field could play an important role in the formation of Sm-Co hard magnetic phase during their crystallization process. In this paper, the magnetic field heat treatment was carried out at different temperatures on as-milled SmCo₅/ α -Fe nanocomposite magnets with different Fe content fabricated by high energy ball milling. The influences of magnetic field heat treatment on their magnetic properties and structures were investigated systematically.

2. Experiment

SmCo₅ ingots were prepared by arc melting. An excess 8 wt.% of Sm over the stoichiometric composition was added to compensate for the losses during melting. The oxygen and nitrogen analyzer and inductively coupled plasma emission spectrometer (ICP) were used to analyze the oxygen content and composition of SmCo₅ ingots, respectively. The results showed that the oxygen content of the ingot is less than 100 ppm and the actual composition is SmCo_{4.96}. SmCo₅ powders with a size less than 150 μ m obtained by manual grinding were mixed with commercial iron powders with a particle size about 10 μ m. 10 or 20 wt.% of total mass was Fe powders. The mixed powders were ball milled for 5 h with a ball to powder ratio of 20-25:1. The amorphous powders obtained by high energy ball milling were firstly pressed into cubes in the glove box under Ar atmosphere, and then pressed at 150 MPa under cold isostatic pressure. The bulk samples were sealed in a quartz tube with a vacuum of 2 \times 10 $^{-5}$ Pa and were heated treatment with a home-made equipment consisting of a heating device and an annular permanent magnet which can provide a stable magnetic field of 2.8T.

Powder X-ray diffraction (XRD) data were recorded at room temperature for the samples obtained at the different preparation process. The crystallization behavior of the as-milled samples was characterized by DSC curve. All the magnetic measurements were performed on a vibrating sample magnetometer (Lakeshore 7410). To get a better comparison, samples without a magnetic field heat treatment was magnetized at an applied field of 2.8T before the magnetic measurements.

3. Results and discussions

The XRD patterns collected at room temperature of the samples before and after ball-milling for 5 h are shown in Fig. 1. The XRD patterns of $SmCo_5$ powders before ball milling show a single phase with $SmCo_5$ -type structure as shown in Fig. 1 (a). The disappearance of diffraction patterns for the mixed iron and $SmCo_5$ sample after ball milling for 5 h indicates the amorphous state of as-milled sample (as seen in Fig. 1)(b). And the re-appearance of Fe and



Fig. 1. The XRD patterns of (a) Sm–Co powders before milling, (b) as-milled SmCo₅ + 20 wt.% α -Fe sample, (c) SmCo₅ + 20 wt.% α -Fe sample milled for 5 h and subsequent annealed at 500 °C for 1 h under a magnetic field.

Sm—Co peaks after a heat treatment at 500 $^{\circ}$ C for 1 h is evident in the XRD patterns as shown in Fig. 1(c). The broader peaks suggest the formation of nanocrystallines after the heat treatment.

The crystallization behavior of as-milled samples was characterized by the DSC curves, which is shown in Fig. 2. It is utilized to guide the heat treatment. It is clear that the crystallization temperature of Sm–Co phase is below 500 °C and not influenced so much by the Fe content.



Fig. 2. The DSC curves of as-milled $SmCo_5 + xwt.\%$ (x = 20,30) samples.

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