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Microstructures and magnetic properties of cast alnico 8 permanent magnets under various heat treatment conditions

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ABSTRACT

There is a close relationship between the magnetic properties of alnico permanent magnetic alloys and the nano-scaled spinodally decomposed structure which results from various heat treatment cycles. In this paper, different heat treatment cycles are employed to cast alnico 8 alloys with composition 32.3Fe-37.5Co-13.7Ni-6.2Al-5.8Ti-3.4Cu-0.2Zr-0.1S-0.8Nb. It is observed that magnetic field treatment at high temperature for specific time is the most effective method to obtain better microstructure and magnetic properties. The mosaic structures consisting of Fe-Co phase and Al-Ni rich phase are best separated during thermomagnetic treatment for 4–5 min and refined during low temperature treatments. The bias growth of the ferromagnetic phase does not develop in absence of magnetic field, and hence the phases are not refined and separated completely. This produces isotropic alnico alloys with low magnetic properties. However, continuous cooling of the alloys in magnetic field followed by isothermal treatment without magnetic field provides moderate magnetic properties. Treatment at high temperatures in field and without field for longer time leads to the coarsening of spinodal phases and poor magnetic properties. The optimum magnetic properties of H_{cj} = 1.7 kOe, B_r = 8.0 kGs and $(BH)_{max}$ = 5.02 MGOe are attributed to the refined microstructure of the thermomagnetically treated alloys.

1. Introduction

Alnico alloys consisting of transition metals are important permanent magnetic materials which have the capability to operate at temperatures above 500 °C [1]. Alnico alloys were extensively researched and produced during 1930s–1960s until they were dropped out of favor due to the discovery of high coercivity Rare Earth (RE) based SmCo and then NdFeB magnets. The high prices and supply restrictions of rare earth (RE) ignited renewed interest in alnico alloys during the last few years [2]. The world wide availability of the main constituent elements of alnico (Al, Ni, Co and Fe) negates monopoly over production. The magnetic properties of alnico alloys are associated with the nanostructured ferromagnetic Fe-Co rich phase implanted in paramagnetic Al-Ni matrix phase. The Fe-Co rich phase is called α_1 while Al-Ni rich phase is called α_2 phase. This two phased structure is formed by spinodal decomposition (SD) process [3,4]. The constituent elements of alnico alloys coexist at high temperature above 1250 °C. However, a miscibility gap develops between the constituent elements when the molten alloy is cooled below 1250 °C. This miscibility gap results in paramagnetic Al-Ni phase and ferromagnetic Fe-Co phases, and the process of splitting is termed as spinodal decomposition [5,6]. These phases are further refined by different heat treatments at various temperatures depending on composition [6]. The chemistry, structure, size and orientation of these two phases play important roles in inculcating magnetic properties in the alloys. Various dopant elements have been added to improve the microstructure and magnetic properties of alnico alloys [7–15]. Different processing conditions, i.e. cooling rate, magnetic field during cooling, holding time in magnetic field, and magnetic field strength have important roles in obtaining magnetic properties in the alloys [8,16]. The heat treatment cycles for alnico 8 include homogenization at around 1250 °C for 30–60 min. Homogenization modifies and fixes the intermetallic structures present at the interfaces, removes the stresses of as-cast alloys, and develops a steady state of chemistry in the alloys [17]. At 1250 °C the homogenized samples exhibit a bcc structure which is locked in the alloys by quenching in water, oil or air. Fast quenching bypasses the formation of deleterious effects of γ (soft magnetic) phase [6]. The homogenized and quenched samples are further treated either with magnetic field or without

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magnetic field. Magnetic heat treatment results in anisotropic alnico magnets while heat treatment without magnetic field results in isotropic alnico magnets. The magnetic heat treatment develops spinodal phases by the textured preferential growth of ferromagnetic phase parallel to the direction of the applied field [18]. Cooling in magnetic field helps to separate ferromagnetic and paramagnetic phases. Different alloy compositions have different suitable temperatures for evolution of desirable microstructures and magnetic properties. Nevertheless, many reports agree that magnetic field treatment for alnico alloys lies between 790 and 860 °C [7,8,16,19]. After the high temperature (1250 °C and above 790 °C) treatments, two low temperature treatments are applied to refine the spinodal phases. These treatments include step aging I at around 650 °C, and step aging II at around 550 °C. The atomic migration is completed during the low temperate treatments through the migration of Fe-Co atoms towards α_1 phase and Al-Ni and other elements toward α_2 phase. The higher aspect ratio (diameter to length ratio) of the ferromagnetic phase during step aging I results in the increment of coercivity. The packing density (the density of Fe-Co rich rods) also increases during aging treatments [20,21]. Both aging treatments are reported to reduce the T_C of Al-Ni rich phase by purifying it from Fe content, so that it behaves as paramagnetic phase at room temperature [11]. Over aging has been reported to have detrimental effects on magnetic properties of alnico alloys [22].

Zhou et al. [16] studied the effect of different heat treatments on alnico 8 alloys produced by powder metallurgy. However, the powder metallurgy, involving close-coupled gas-atomization and complicated hot isostatic pressing (HIP), is a difficult and expensive fabrication technique. Furthermore, complex shaped alnico magnets (end products) can be best made by casting techniques. In this paper, we report the effects of different heat treatments on the microstructure and magnetic properties of alnico 8 alloys produced by a rather simple method, i.e., the traditional casting and simplified but effective heat treatment techniques. The effects of different heat treatments (with and without magnetic field) on microstructure and magnetic properties are studied and compared. We have tried to establish a close relationship between nano-scaled spinodally decomposed structure and magnetic properties of cast alnico 8. Discussed also are the effects of different inclusions and their possible roles in the alloy.

2. Experimental details

Alnico alloys were produced by melting in electric arc furnace equipped with magnetic stirrer and casting technique using pure elements (99.99 wt%) of nominal composition 32.3Fe-37.5Co-13.7Ni-6.2Al-5.8Ti-3.4Cu-0.2Zr-0.1S-0.8Nb. After melting the alloys composition was verified by XRF analysis. Cylindrical samples of 5 mm in diameter and 5 mm in length were obtained from the cast ingots by electric discharge machining. Various heat treatments were applied after homogenization at 1250 °C for 35 min. In the first experiment, samples were furnace cooled from 855 °C to 810 °C at the rate of 5 °C/min and treated at 810 °C for 30 min in Ar. atmosphere. The samples were further cooled down to 650 °C at the rate of 8 °C and treated 5 h at 650 °C (here after HT-I) and 15 h at 540 °C (here after HT-II). Another group of samples were furnace cooled from 855 °C to 840 °C and treated for 30 min at 840 °C followed by HT-I and HT-II. In the second experiment samples were air cooled (~60 °C/second) in magnetic field (0.6 T) from 1250 °C to 810 °C and treated at 810 °C for 30 min without magnetic field followed by HT-I and HT-II. Another group of samples were air cooled down from 1250 °C to 840 °C in magnetic field and treated at 840 °C for 30 min without field, followed by HT-I and HT-II. In the last experiment samples were treated at 840 °C for longer time (2–30 min) in magnetic field followed by HT-I and HT-II. The samples were etched in 7 ml HNO_3 and 100 ml ethanol solution for 40–50 s and studied under Olympus measuring microscope, STM 7 and field emission scanning electron microscope (FE-SEM, MLA650F) for microstructural analysis. The crystalline structure was determined by X-ray diffractometer using Cu kα radiation.

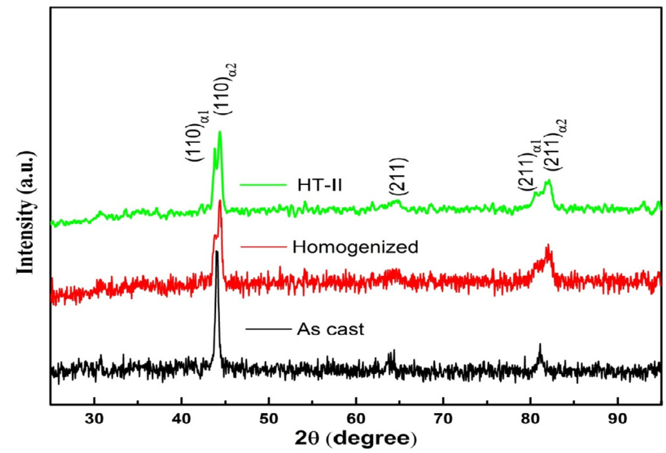

Fig. 1. X-ray diffraction patterns of alnico alloys at different stages of heat treatment.

3. Results and discussion

Fig. 1 shows the XRD patterns of the designated alnico alloys at different stages of heat treatment. The peaks observed in the patterns are compared with PDF card no. 06–0696. The as-cast and homogenized alloys exhibit bcc structure with peaks at 44.3 and 82.2° 2θ corresponding to (110) and (211) indices. The pattern of the cost alloy has sharp peaks while the peaks in homogenized alloy are broadened, which indicates that the spinodal phases have started to separate during quenching process. The peaks of the fully heat treated (HT-II) alloy have split into two parts, which indicates that the bcc α phase has separated to form α_1 and α_2 phases during heat treatment. The lattice parameters are slightly deviated from bcc towards tetragonal structure (a/c = 0.99 and c/a = 1.007) due to higher content of Co. These results are in agreement with the findings of Liu et al. [11] who showed that the bcc structure of alnico becomes slightly tetragonal due to high content of Co.

The magnetic properties of alnico 8 alloys at different stages of heat treatment are given in Table 1. The as-cast alloys have low intrinsic coercivity, H_{cj}, while partial decomposition has taken place during air quenching. The H_{cj} of the alloy is enhanced when cooled from 855 °C to 810 °C at the rate 5 °C/min and annealed at 810 °C for 30 min. The cooling rate and annealing temperature have important effects on the magnetic properties of the alloys. Cooling from 855 °C to 840 °C and annealing at 840 °C does not result in good magnetic properties. The magnetic properties were improved when the cooling was performed in magnetic field and treated at 840 °C and 810 °C. Continuous cooling in magnetic field is usually performed for commercial alnico 5 alloys [21]. However, it is also useful in alnico 8 alloys for economic considerations. It is reported that treatment above T_C leads to the growth of soft magnetic phases in the alloys, while activation energy of atoms is not enough for migration of atoms below 790 °C. This makes spinodal decomposition process sensitive to both lower and upper temperatures [9]. To further improve the magnetic properties, the samples were treated in magnetic field for various times at 840 °C and 810 °C. The treatment at 840 °C was found out to be the optimized temperature for improving magnetic properties. Annealing time is also critical to enhance magnetic properties. The magnetic properties increase initially as a function of magnetic annealing time, reach a saturation point and finally decrease. For the current investigated alloys, the best annealing condition is found to be 4–5 min in magnetic field at 840 °C, followed by HT-I and HT-II. The magnetic field annealing at 810 °C also yields good magnetic properties, but it takes longer time as compared to