Journal of Magnetism and Magnetic Materials 321 (2009) 325–329

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

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

Texture, microstructure and magnetic properties of Fe–28Cr–15Co–3.5Mo permanent magnet

Z. Ahmad, A. ul Haq *

Institute of Industrial Control System, P.O. Box 1398, Rawalpindi, Punjab 44000, Pakistan

ARTICLE INFO

Article history:
Received 23 April 2008
Received in revised form
4 September 2008
Available online 18 September 2008

Keywords:
Texture
Microstructure
Anisotropy
Magnetic property
Fe–Cr–Co–Mo permanent magnet

ABSTRACT

Crystallographic texture, microstructure and magnetic properties of Fe–28Cr–15Co–3.5Mo alloy were studied as a function of processing parameters. Texture studies revealed that thermo-magnetic aging leads to the development of ideal Goss type, {1 1 0} ⟨0 0 1⟩ and cube type {0 0 1} ⟨0 1 0⟩ textures. The orientation densities of these texture components become stronger after the step-aging treatments. Microstructural features show that improvement in magnetic properties were due to aligning and elongation of ferromagnetic Fe, Co-rich (α_1) particles in the preferred ⟨1 0 0⟩ directions. Magnetic analysis reveals that magnetic properties of the alloys are directionally dependent and influenced by the choice of thermo-magnetic treatment temperature or time. The maximum values of intrinsic coercive force, remanence and energy product, obtained in the textured magnetic alloy were 68.99 kA/m (867 Oe), 1.12 T (11.2 kG) and 43.2 kJ m^3 (5.4 MG Oe), respectively.

1. Introduction

The Fe–Cr–Co permanent magnets possess excellent properties including ductility, corrosion resistance, high $(BH)_{max}$, high remanance and have comparable magnetic properties with those of Alnico magnets [1–3]. They have wide range of applications in magnetic sensors, telephone receivers, printers, stereo-cartridges, hysteresis motors, aircraft magnets, etc. [3,4]. Permanent magnetic properties in Fe–Cr–Co alloys are resulted from spinodal decomposition; whereby high-temperature bcc phase α breaks down into two spinodal phases known as Fe, Co-rich α_1 and Cr-rich α_2 that are stabilized by annealing the alloy in the temperature range 560–650 °C [5,6] depending upon the composition of the alloy. Permanent magnets based on Fe–Cr–Co alloy system were discovered in 1972, by Kaneko et al. [7]. The heat-treatment process used in their studies was solution annealing between 1300 and 1350 °C in argon, followed by rapid quenching, 30 min spinodal aging at 630–640 °C in a magnetic field, and a final heat treatment without a field for up to 6 h at 600 and 560 °C. The best properties obtained were $H_c = 46$ kA/m (580 Oe), $B_r = 1.3$ T (13 kG) and $(BH)_{max} = 42$ kJ/m^3 (5.4 MG Oe) with 45Fe–30Cr–25Co (wt%) alloy.

The anisotropic Fe–Cr–Co-based bulk magnets are produced by the magnetic field aging process, while magnets in thin sheet or wire shape are obtained by the deformation aging technique [4,8]. Deformation aging produced magnetic thin sheets, rods or wires have produced maximum magnetic properties as $H_c = 85.8$ kA/m (1080 Oe), $B_r = 1.3$ T (13 kG) and $(BH)_{max} = 79$ kJ m^3 (9.8 MG Oe) in 42Fe–33Cr–23Co–2Cu (wt%) alloy [9], but these are expensive due to involvement of long processing cycle which put restriction in their practical applications. Previous studies emphasize the magnetic properties in high Co-containing Fe–(26–37)Cr–(16–25)Co alloys. Later, the studies were shifted to low cobalt-containing Fe–(22–37)Cr–(6–15)Co alloys, as Co is expensive as well as strategic. Efforts were made to improve magnetic properties with low Co-containing alloys by addition of α former element like Mo, V, Al, Ti, Si, W as well as optimizing the processing conditions. As an example, Mo addition to the ternary Fe–(22–30)Cr–(15–18.5)Co alloys is found to increase the magnetic properties due to aligning the spinodal phases α_1 and α_2 along ⟨1 0 0⟩ directions [10,11]. It means that good magnetic properties with Fe–Cr–Co–Mo alloys can be obtained by aligning and elongating the spinodal phases in ⟨1 0 0⟩ texture samples. This was done with Fe–22Cr–18.5Co–Mo single crystal alloy and the best magnetic properties were obtained as $H_c = 72.4$ kA/m (910 Oe), $B_r = 1.58$ T (15.8 kG) and $(BH)_{max} = 90.74$ kJ m^3 (11.4 MG Oe) [12]. Similarly, energy product up to 75.6 kJ m^3 (9.5 MGOe) were also reported by developing ⟨1 0 0⟩ columnar grain structure using the chill casting method with Fe–24Cr–15Co–3Mo–1Ti alloys [13]. But the process to develop single crystals and ⟨1 0 0⟩ columnar grain structure is quite expensive due to a long heat-treatment cycle. Szymura et al. [14] have developed (1 1 0) ⟨0 0 1⟩ texture by cold rolling and recrystallization process in strips (0.5 mm thin) of Fe–30Cr–14Co–3Mo alloy. Sugimoto and

* Corresponding author. Tel.: +92 51 9268130; fax: +92 51 4479597.
E-mail address: anwar.lhq@gmail.com (A. ul Haq).

0304-8853/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jmmm.2008.09.007

co-workers [2] have produced {1 1 0} ⟨1 1 0⟩ and {1 1 0} ⟨1 0 0⟩ texture in Fe–30Cr–15Co–3Mo alloys by the combination of annealing and recrystallization processes. The best magnetic properties reported in their work were $H_c = 82$ kA/m (1025 Oe), $B_r = 1.2$ T (12 kG) and $(BH)_{max} = 56$ kJ m^3 (7.0 MG Oe). The literature indicates that previous studies were conducted to enhance magnetic properties of Fe–Cr–Co–Mo alloys but economical means to produce Fe–Cr–Co–Mo bulk magnets have not been investigated systematically so far.

The purpose of the present work is to

(a) develop anisotropic Fe–28Cr–15Co–3.5Mo bulk magnets in comparison with Alnico magnets,
(b) optimize the processing parameters,
(c) establish economical processing cycle,
(d) investigate the development of texture, microstructure and magnetic properties produce during processing of alloy.

2. Experimental

The chemical composition of the studied alloys was (in wt%) Fe: 43.0 ± 0.1, Cr: 28.0 ± 0.1, Co: 15.0 ± 0.1, Mo: 3.50 ± 0.11, C: 0.001, S: 0.001, N: 0.0176. The alloy composition was verified by the wet method using the inductively couple plasma (ICP) technique, and the carbon, sulphur and nitrogen contents were determined using LECO devices for these elements. Cast ingots of 85 mm in diameter and 130 mm in length were prepared by vacuum induction melting using 99.9% pure elements. Ingots were homogenized at 1250 °C for 48 h and then hot rolled into bars of 23 mm diameter at 1200 °C. Square samples of 10 mm side and 7 mm in thickness were cut from the hot-rolled bars and subjected to heat treatments in an argon atmosphere. The heat treatment of the alloy was composed of solution treatment, thermo-magnetic treatment (TMT) and step-aging treatment. Square samples were solution treated for 30 min at 1250 °C followed by fast quenching into ice water. The TMT was performed in two steps. In the first step, solution-treated samples were reheated for 40 min in the temperature range 620–650 °C and quenched into water (TMT-1). In the second step, TMT-1 samples were heat treated for 1–5 h in the temperature range 600–630 °C and quenched into water (TMT-2). Both TMT-1 and TMT-2 treatments were carried out under the axial magnetic field strength of 2.5 kOe. During the step-aging treatment, TMT samples were heated at 605 °C for 3 h, then cooled at the rate of 3 °C per hour to 490 °C and held for 8 h and finally furnace cooled.

Texture studies were carried out by plotting pole figures as well as the orientation distribution function (ODF) which describes the crystallite orientation densities in a three-dimensional orientation space defined by the Euler angles (φ_1,φ,φ_2). For this purpose from each sample, three incomplete pole figures, {1 1 0}, {2 0 0} and {2 1 1}, were measured by the Schulz reflection method [15] using CoKα radiation on a four-circle texture goniometer. The pole figures were measured up to a maximum tilt angle of 70° in steps of $\alpha = 5°$ and $\beta = 3.6°$. From these pole figures, the ODF was calculated using a series expansion method [16] up to $L_{max} = 22$. For all samples, 45°-ODF sections were shown as they represent the most prominent texture for bcc and fcc structures of the materials [17].

Microstructural examination was carried out on an optical microscope and a scanning transmission electron microscope attached with an energy dispersive spectrometer (EDS) to facilitate the chemical composition analysis. Sample preparation for optical and electron microscopy is described elsewhere [11]. Magnetic properties were measured using ±10 kOe maximum applied field on a magnetometer after calibrating with an Alnico-5 standard sample.

3. Results and discussion

3.1. Microstructural examination

Fig. 1 shows the microstructure of the alloy obtained in the solution-treatment state. The optical microscopy shows that the microstructure is composed of coarse grains of α-phase along with non-metallic inclusions in the form of black spots. The average grain size was measured by the linear intercept method as 980 ± 5 μm. The SEM micro chemical analysis shows the inclusions which are rich in chromium (85–90 wt% Cr). Since this technique cannot detect the light elements, such as nitrogen or carbon, the actual composition may not be ascertained. However presence of high value of nitrogen impurity (0.00176 wt% N) in the alloy suggests that the inclusions may be identified as Cr_2N. The volume fraction of this compound was too small to be detected by the X-ray diffraction technique. Thus, the X-ray diffraction analysis indicated only presence of bcc α phase after the solution-treated state.

Fig. 2 shows the microstructure and the corresponding electron diffraction pattern of the magnetic alloy after step-aging treatment. The bright rod-like particles in the micrographs were identified as α_1 phase (highly magnetic) embedded in dark α_2 phase (weakly magnetic) [5]. The microprobe analysis revealed that Fe, Co are preferentially segregated in the α_1 phase and Cr, Mo are preferentially segregated in the α_2 phase. The magnetically rich α_1 particles were 18–25 nm in length and seem to be preferentially aligned and elongated in the applied field direction as marked with the arrow (Fig. 2). The volume fraction of α_1 particles in the alloy was ~37%. The microstructural studies suggest that better magnetic properties in the studied alloys are expected due to better shape anisotropy of the α_1 particles.

3.2. Texture studies

In Fig. 3, the 45°-ODF sections of solution-treated, TMT and step-aged samples are compared. The solution treatment has produced a major {1 0 1} ⟨2 3 2⟩ texture component at $\varphi_1 = 45°$, $\varphi = 45°$ and $\varphi_2 = 90°$ with 1.97× random orientation density and a minor cube {0 0 1} ⟨0 1 0⟩ texture component with 1.30× random orientation density (Fig. 3a). The {1 0 1} ⟨2 3 2⟩, {0 0 1}

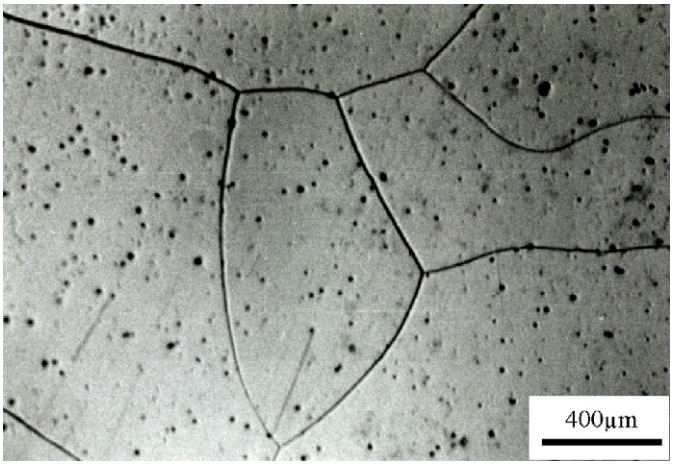

Fig. 1. Micrograph showing coarse α grains following the solution treatment at 1250 °C for 30 min.