Journal of Magnetism and Magnetic Materials 402 (2016) 115–123

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

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

Magnetic characterization of nanocrystalline $Fe_{14}Nd_2B_1$ alloy during melt spinning and subsequent annealing

S. Sarafrazian, A. Ghasemi, M. Tavoosi *

Department of Materials Engineering, Malek-Ashtar University of Technology (MUT), Shahin-Shahr, Isfahan, Iran

ARTICLE INFO

Article history:
Received 12 August 2015
Received in revised form
17 November 2015
Accepted 19 November 2015
Available online 1 December 2015

Keywords:
Amorphous/Nanocrystalline
$Fe_{14}Nd_2B_1$ Alloy
Melt spinning
Magnetic material

ABSTRACT

The magnetic characterization of amorphous/nanocrystalline $Fe_{14}Nd_2B_1$ alloy during melt spinning and subsequent annealing was the goal of this study. The melt spinning process was done at different wheel speeds in the range of 20 to 40 m s^{-1}. To achieving the desired microstructure, the annealing process was also done in melt spun ribbons at temperature range of 500 to 700 °C for different periods of time. The melt spun and annealed samples were characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), dispersive X-ray spectrometry (EDS), differential scanning calorimetry (DSC) and vibrating scanning magnetometer (VSM). According to achieved results, the microstructure of melt spun ribbons were combination of $Nd_2Fe_{14}B$, Fe-α and amorphous phases with the coercivity and saturation of magnetization in the range of 11.2–125.6 kA/m and 65–120 A m^2/kg, respectively. By annealing the ribbons, the coercivity and saturation of magnetization increased and decreased, respectively. The highest values of coercivity (752 kA/m) and stored magnetic energy (about 267.68 kJ/m^3) were achieved in annealed sample at 600 °C for 6 h.

1. Introduction

Nd–Fe–B hard magnetic alloys were commercialized due to their high coercivity and low magnetic permeability [1–3]. These magnets have become an integral part of many electrical components since their development in 1984 [4,5]. The microstructure of these alloys is a combination of hard (such as $Fe_{14}Nd_2B_1$) and soft (such as Fe-α, Fe_3B) magnetic phases. In fact, the magnetic properties of Nd–Fe–B alloys directly depend on the crystallite sizes, volume percentage and distribution of hard and soft magnetic phases in microstructure.

The crystallization of amorphous precursors is the effective method for the formation of the desired microstructures with optimum magnetic characterization in Nd–Fe–B alloys [6,7]. For instance, Inoue et al. [8] prepared a Fe/$Fe_{14}Nd_2B_1$ nanocomposite microstructure with optimum magnetic properties by crystallization of $Fe_{89}Nd_7B_4$ amorphous melt spun ribbons. Nagase et al. [9] also presented one microstructure with best magnetic properties in $Fe_{14}Nd_2B_1$ compound by means of melt spinning and annealing processes.

Although there are a lot of investigations about the formation and magnetic characterization of amorphous/nanocrystalline $Fe_{14}Nd_2B_1$ alloy [8,9], no optimum annealing condition for achieving the best magnetic properties in this alloy has not been presented so far. Therefore, optimization of the melt spinning and annealing process of $Fe_{14}Nd_2B_1$ alloy in order to achieve the best magnetic properties was the goal of this study.

2. Materials and methods

A master alloy with a nominal composition of 82.36% Fe–11.76% Nd–5.88% B (in at%) was prepared by standard argon arc melting of 99.5% purity Fe, Nd and B elements (all sample preparation were done in glove box under argon atmosphere). The produced alloy was re-melted three times to ensure homogeneous mixing. The melt-spun ribbons were produced by injecting the melt onto a rotating copper wheel of 240 mm diameter with substrate velocities in the range of 20–40 m s^{-1} in argon atmosphere and a quartz crucible orifice of 0.6 mm. The pressure of spinner chamber before melt injection was about 5×10^{-3} Pa which increased to 10^5 Pa during melt spinning process. Heat treatment procedure had been done at temperatures range of 500–700 °C at different periods of time until 8 h. Before annealing, the as-spun ribbons were sealed in a quartz tube under the vacuum of 10^{-3} Pa in order to prevent form oxidation during annealing.

An XRD using a diffractometer with Cu Kα radiation (λ=0.15406 nm; 40 kV; Philips PW3710) was used to follow the structural variation of the specimens (2θ range: 20–80°, step size: 0.05°; time per step: 1 s). The crystallite sizes and volume fraction

* Corresponding author.
E-mail address: ma.tavoosi@gmail.com (M. Tavoosi).

http://dx.doi.org/10.1016/j.jmmm.2015.11.052
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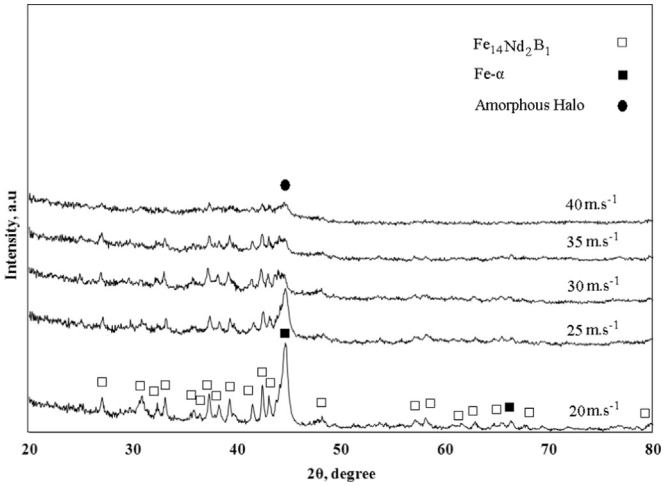

Fig. 1. The XRD patterns of the melt spun $Fe_{14}Nd_2B_1$ alloy quenched at different wheel speeds.

of $Fe_{14}Nd_2B_1$ and Fe-α phases estimated form XRD patterns using Scherrer and internal standard methods [10], respectively. Structural and morphological characterizations of samples were carried out by field emission scanning electron microscopy (VEGA-TESCAN-XMU) at an accelerating voltage of 20 kV which was equipped with detector for energy dispersive X-ray (EDS) analyses. Differential scanning calerimerty was also conducted to study the thermal stability of produced amorphous alloy using the L81/1750 DTA differential thermal analyzer. The samples were placed in Al_2O_3 pans and heated in dynamic argon atmosphere up to 800 °C at a heating rate of 20 °C/min. Magnetic properties of produced samples were measured using a vibrating scanning magnetometer under an applied field up to 1200 kA/m. About 0.025 g of produced ribbons (without any crushing or milling) was directly placed in the sample holder and the test was started. Moreover, the Gibbs free energy changes of formation of amorphous phase in $Fe_{14}Nd_xB_{3-x}$ $(0 \le x \le 3)$ alloys were estimated according toMiedema's model [11,12].

Table 1
The crystallite sizes of $Fe_{14}Nd_2B_1$ and Fe-α phases melt spun $Fe_{14}Nd_2B_1$ alloy quenched at different wheel speeds.

	Crystallite size (nm)				
Wheel speed (m s^{-1})	**20**	**25**	**30**	**35**	**40**
$Fe_{14}Nd_2B_1$	20	12	7	5	3
Fe-α	30	15	6	-	-

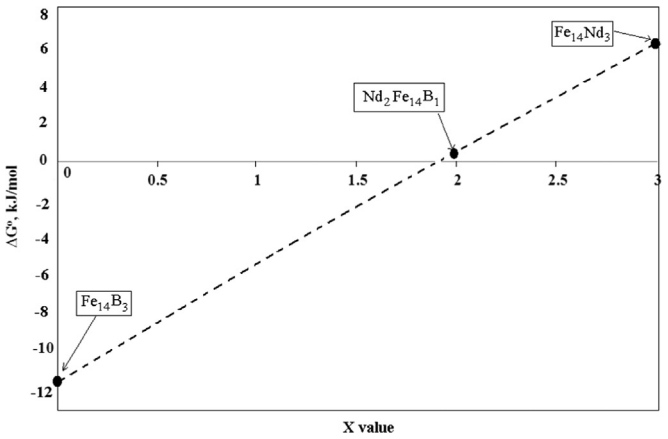

Fig. 3. The Gibbs free energy changes of formation of amorphous phase in $Fe_{14}Nd_xB_{3-x}$ $(0 \le x \le 3)$ alloys (according to Miedema's model).

3. Results and discussion

3.1. Melt spinning process

There are several factors such as solidification rate, superheat, melt spinning atmosphere, gap distance between nozzle and wheel and wheel surface quality that influence the cooling behavior and the structure and properties of the rapidly solidified alloy [4,5]. A slight variation in the process parameters can often cause large variations in the microstructure and properties of the produced samples. In the

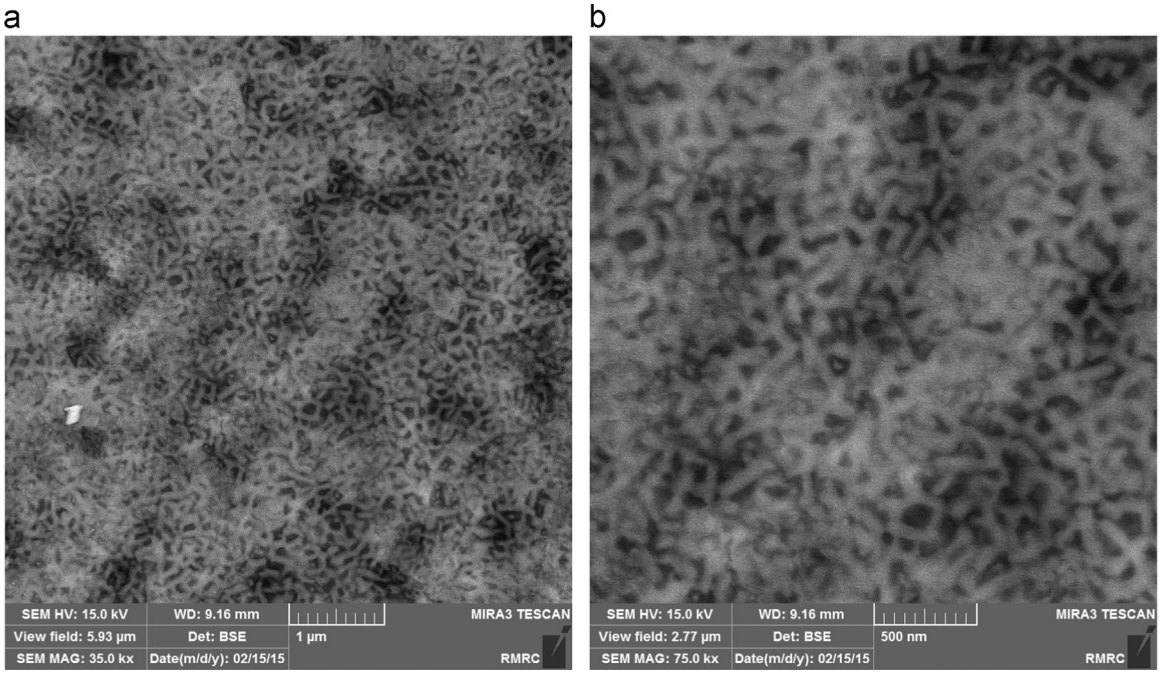

Fig. 2. The FE-SEM micrographs of melt spun $Fe_{14}Nd_2B_1$ ribbons at wheel speed 20 m s^{-1} in two magnifications.