Production and Characterization of Nano Crystalline $Fe_{85}Si_{10}Ni_5$ Soft Magnetic Alloys by Mechanical Alloying

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ARTICLE INFO

Article history:

Received 24 Jun. 2014
Accepted 27 Aug. 2014
Available online 01 Dec. 2014

Keywords:

Nano crystalline materials
Mechanical alloying
Soft magnetic powders
Magnetic properties

ABSTRACT

There are various methods to produce iron based nano crystalline magnetic alloys. Among these methods, mechanical alloying is one of the most important. In this research, nano crystalline $Fe_{85}Si_{10}Ni_5$ soft magnetic alloy was synthesized by mechanical alloying. The effect of alloying time on phase constituents and magnetic properties of the produced powders was investigated, by X-ray diffraction (XRD) and alternating gradient force magnetometer (AGFM). The XRD results showed that alloy formation started after 2hours of milling. Further milling resulted in the reduction of the grain size and lattice parameter. After 60 hours of milling, the grain size was reduced to 8 nm. AGFM results showed that magnetic saturation and coercivity depends on the alloying time. Increasing the alloying time, causes the increase of magnetic saturation and decrease of coercivity.

1. Introduction

Mechanical alloying is an efficient method in order to produce solid nano crystalline powders in which primary particles continuously undergo an intense plastic deformation and cold weld and fracture [1]. Minimizing the grain size to the scale of nano meter during mechanical alloying will cause main changes in physical properties, especially magnetic properties of materials. Although decreasing the grain size will cause stress and defects in the structure of materials, some of their magnetic properties will be improved generally. Therefore, magnetic properties of materials would be in contradiction with decrease of grain size and increase of structure defects and stresses [2].

Iron based nano crystalline soft magnetic alloys have been considered since the end of the 80s due to their favorable magnetic properties. Such alloys have found wide application in information storage, generators, transformers and magnetic cores [3]. Nano crystallinemagnetic powders of FeSiNi are used greatly in magnetic cores due to their ability in elimination of electromagnetic noises [4].

Although pure iron is considered as a ferromagnetic material, it will cause a great eddy current loss because of its low electrical resistance [5]. Therefore, addition of some elements such as Si and Ni to pure iron will cause increase of its electrical resistance and subsequently decrease of eddy current loss. Besides, addition of Si will cause reduction in magnetic anisotropy and coercivity. Likewise, Ni would be an agent for increasing the permeability and saturation magnetization [2]. This research aims to investigate the possibility

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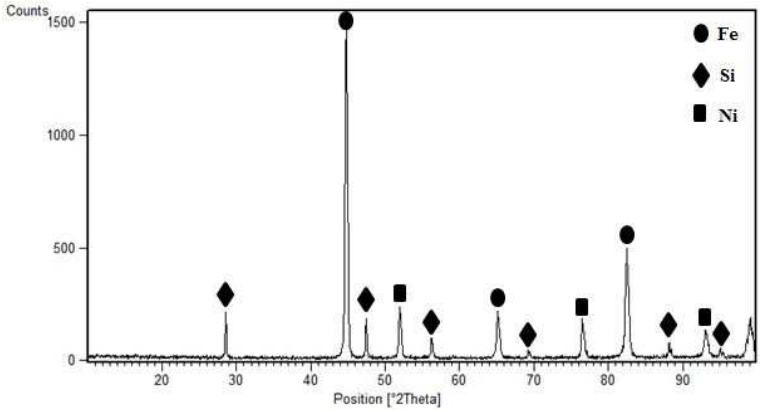

Fig. 1. X-ray diffraction pattern of the $Fe_{85}Si_{10}Ni_5$ powders forinitial milling times

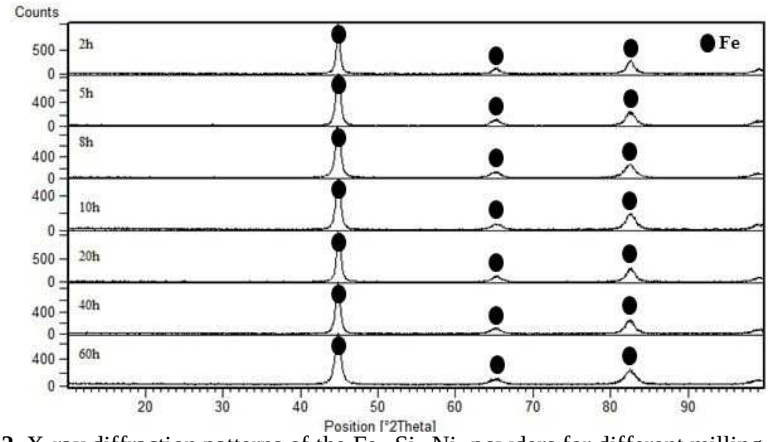

Fig. 2. X-ray diffraction patterns of the $Fe_{85}Si_{10}Ni_5$ powders for different milling times

of producing soft magnetic powder of $Fe_{85}Si_{10}Ni_5$ by mechanical alloying and to examine the effects of milling timeon their physical and magnetic properties.

2. Experimental

In this research, mixtures containing 85 wt% of Fe (with 99% purity and10 µm average particle size), 10 wt%of Si (with 99% purity and 10 µm average particle size) and 5 wt% of Ni (with 99% purity and 10 µm average particle size) were prepared and then ball milled in different times in argon atmosphere (with 99.99% purity) using a ball mill (FP4) made of a chromium alloyed steel cylindrical container comprised of five balls (diameter: 20 mm). The ball-to-powder weight ratio and the speed were chosen as 20:1 and 600 rpm, respectively, and the mixtures were milled for 0,2,5,8,10,20,40 and 60 hours. X-ray diffraction (XRD, Phillips X'pert, using $Cu_{K\alpha}$ with $\lambda_{Cu\ K\alpha}$ =1.5406 ºA) was applied for phase analysis of the milled powders. The lattice parameter was measured by Bragg's law, and lattice strain and the crystallite size of the milled particles were measured by Williamson - Hall method. In order to study the magnetic characteristics, powders milled for 2, 5, and 20 hours were selected and then characterized by the alternating gradient force magnetometer (AGFM) in room temperature and a magnetic field of 9000 Oersted (Oe).

3. Results and discussion

X-ray diffraction patterns are shown in figures 1 and 2. As can be seen, the peaks of Si and Ni have disappeared after two hours of milling, which displays diffusion of Ni and Si into Fe and the start of formation of a solid solution. Increasing the milling time up to 60 hours has

Table 1. Changes of lattice parameter, grain size, and lattice strain vs. the milling time

Time (hours)	lattice parameter(nm)	Grain size(nm)	Lattice strain (%)
0	0/2867	-	-
2	0/2857	53	0.02
5	0/2861	32	0.03
8	0/2866	26	0.03
10	0/2864	18	0.03
20	0/2862	14	0.03
40	0/2859	10	0.03
60	0/2855	8	0.04

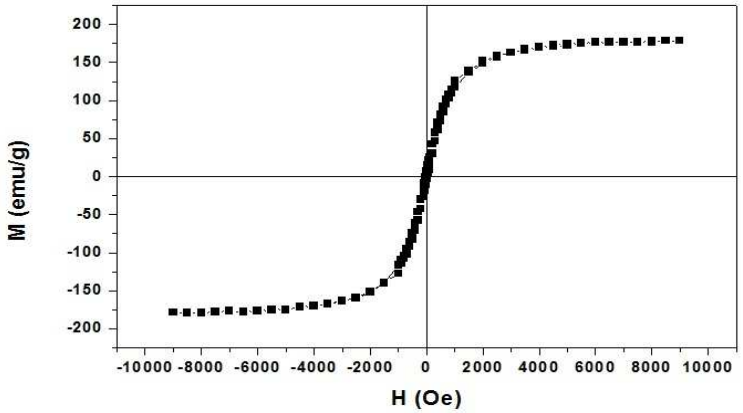

Fig. 3. Residual loop of the $Fe_{85}Si_{10}Ni_5$ powders after 2 hours of milling

caused no changes to the number of the peaks; only their width are broadened and the intensity decreases because of the lattice strains and the decrease of grain size. Mechanical stresses occurred by mechanical alloying process create a high density of dislocations and other crystal defects in the particle powders which result in an elastic deformation in the lattice and changes in the distance between crystal planes which is the result of departure of the atoms from their accidental original locations in the crystal structure. Therefore, one plane with a special miller index and not much different diffraction angles will set with Bragg's equation, which will cause many close peaks belonging to that special plane, so that we will observe a wide peak in the XRD pattern in a special angle. Besides, with reduction of the grain size, the number of crystal planes in a crystallite will decrease. In other words, the number of planes causing destructive interfaces will be reduced, and the angles that do not set with Bragg's equation cannot be eliminated. So, all the Bragg's angles will show diffraction as well and, as a consequence, a wide peak will be observed [6].

Table 1 shows the changes of lattice parameter, grain size, and lattice strain according to the milling time. It can be observed that by increasing the milling time, lattice parameter decreases with a negligible fluctuation from 0/2867 nm (during 0 hour of milling) to 0/2855 (during 60 hours of milling). Decrease in lattice parameter can be counted as a result of dissolution of Ni and Si atoms (with smaller atomic radii than that of Fe) into Fe. However, because the sizes of atomic radius of Fe and Ni are very close, the lattice parameter has decreased very little [7]. Also, as can be seen in Table 1, with increase of the milling time the particles grain size has decreased, which could be due to the work hardening as a consequence of intense plastic deformation of powders during the milling process [8].

Figures 3, 4, and 5 show the residual loops of powder samples after 2, 5, and 20 hours of milling process, respectively, and Table 2 represents coercivity and saturation magnetization measures regarding the residual loops. Coercivity is one of the important factors

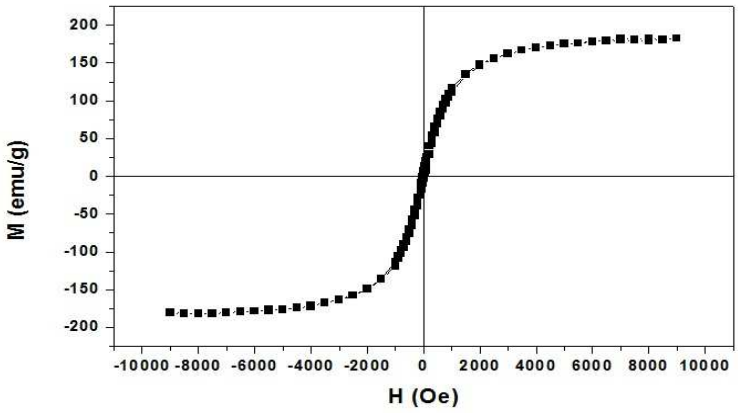

Fig. 4. Residual loop of the $Fe_{85}Si_{10}Ni_5$ powders after 5 hours of milling

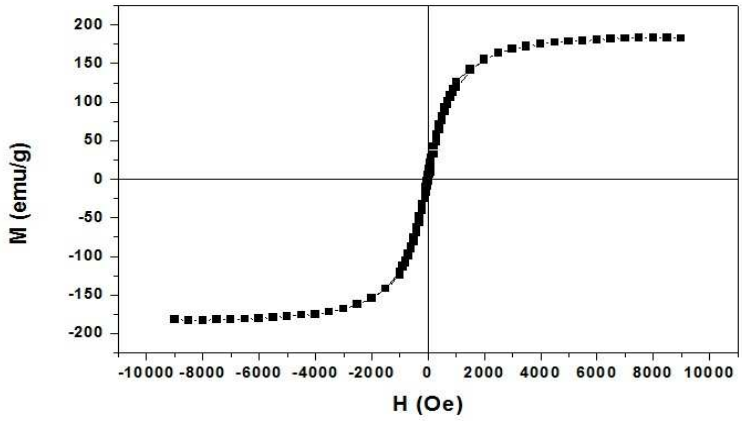

Fig. 5. Residual loop of the $Fe_{85}Si_{10}Ni_5$ powders after 20 hours of milling

Table 2. Changes of coercivity (Hc) and saturation magnetization (Ms) vs. the milling time

Time(hours)	Hc(Oe)	MS(emu/g)
2	36	177
5	34	181
20	30	182

in determining the magnetic behavior of materials. Soft magnetic materials are defined as materials with low coercivity and high and thin residual loops [9]. According to the information mentioned in this Table, the maximum measure of coercivity belongs to the powder sample milled for 2 hours which could be because of incomplete alloying and unfinished dissolution of Si and Ni into Fe. In addition, the grain size is an effective parameter on coercivity; this means that by increasing the milling time and consequently reduction of the crystallite size, coercivity will increase if the crystallite size is more than magnetic exchange length (L_{ex}) which could be concluded from equation (1).

$$Hc = 3\sqrt{\frac{K_B T_C K_1}{a M_S}}\frac{1}{D} \quad [1]$$

Where (D) represents the grain size; (M_S) is saturation magnetization, (K_1)is magneto-crystalline anisotropy, (T_C) is Curie temperature, (K_B) is Boltzmann constant, (a) is lattice constant and (H_C) is coercivity.

L_{ex} could be calculated by equation (2) [8]:

$$L_{ex} = \sqrt{\frac{A}{K_1}} \quad [2]$$

In this equation, (A) is exchange stiffness constant.

By increasing the milling time, the particles grain size will intensely get smaller. If the grain size becomes smaller than L_{ex}, a direct relation would be held between coercivity and grain size according to equation (3) and, as a consequence, coercivity will reduce by decreasing the grain size. Therefore, regarding the information mentioned in Table2 coercivity will decrease by increasing the milling time [8].

$$H_C = \frac{P_C K_1^3 D^6}{\mu_0 M_S A^3} \quad [3]$$

In this equation, (μ_0) is permeability of free spaceand (P_C) is constant of the order of unity.

The quantities of saturation magnetization represented in Table2 show that by increasing the milling time, saturation magnetization increases too. This phenomenon could happen because of completion of the alloying process and decrease of magneto-crystalline anisotropy achieved by fine grain size causing easier rotation of magnetic domain walls [8]. Also by decrease of the crystallite size, each crystallite could be a single magnetic domain which will eliminate the influence of magnetic domain walls and will increase the saturation magnetization. This fact, increase of saturation magnetization by decreasing the grain size, could be considered similar to the increase of mechanical strength due to the decrease of the grain size. So, the grain size has a similar effect on dislocation movement and magnetic domain walls in stress and magnetic fields, respectively [3]. Coercivity of the powder sample milled for 20 hours is in the range similar to that of soft magnetic materials.

4. Conclusions

1. Mechanical alloying using ball mills was an efficient method in order to produce nano crystalline solid solution of Si and Ni in Fe with soft magnetic properties.
2. The grain size and the lattice parameter decreased by increasing the milling time. Therefore, by milling the primal powders of Fe, Ni and Si during 2 up to 60 hours particles with grain size between 53 to 8 nm were obtained.
3. Coercivity and saturation magnetization depends on the milling time. Therefore, by increasing the milling time, coercivity decreased and saturation magnetization increased. The minimum coercivity and the maximum saturation magnetization were obtained for the powder sample milled for 20 hours.

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