Proceedings of the All-Polish Seminar on Mössbauer Spectroscopy, Warsaw, Poland, June 18–21, 2010

Hyperfine Interactions and Some Magnetic Properties of Nanocrystalline $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ Alloys Prepared by Mechanical Synthesis and Subsequently Heat Treated

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$Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ ternary alloys were prepared by mechanical alloying method. To check the stability of their structure thermal treatment was applied subsequently. As X-ray diffraction studies proved the final products of milling were the solid solutions with bcc lattice and the average grain sizes ranged of tens of nanometers. After heating of the $Co_{50}Fe_{45}Ni_5$ alloy up to 993 K the mixture of two solid solutions with bcc and fcc lattices was formed. In other cases thermal treatment did not change the type of the crystalline lattice. Mössbauer spectroscopy revealed hyperfine magnetic field distributions which reflected the different possible atomic surroundings of ^{57}Fe isotopes. Results of the macroscopic magnetic measurements proved that both investigated alloys had relatively good soft magnetic properties.

PACS: 81.20.Ev; 75.50.Kj; 61.10.–i; 76.80.+y; 75.60.Ej

1. Introduction

Co-Fe based alloys are of great interest in current research due to their soft magnetic properties. They attract a lot of attention because of possibility of their application in magnetic mass storage devices [1–5]. All available literature reports are focused on such alloys obtained in traditional way i.e. melting or electrodeposition. We propose mechanical alloying (MA) as the competitive technology of production of magnetically soft Co-Fe-Ni alloys.

MA is a fundamentally different approach to alloy manufacture because it relies on deformation processes to mix materials. It is described as a high energy milling process in which powder particles are subjected to repeated cold welding, fracturing, and rewelding [6]. The MA technology allows alloying of elements that are difficult or even impossible to combine by conventional melting methods. However, different systems react to milling in different ways depending on the mutual reactivity and solubility of the components, their mechanical properties and the type of used milling equipment [6].

The intense deformation applied during milling process can force atoms into positions where they may not prefer to be at equilibrium. Additional heat treatment of the mechanically synthesized alloys may cause structural changes, i.e. an increase in the grain size accompanied by a decrease in the level of internal strains or the change of the crystalline lattice type [7–9]. The aim of this work was: (1) to synthesize $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ alloys by MA technology and (2) to characterize the structure, hyperfine interactions and macroscopic magnetic properties of the alloys after MA process as well as after heat treatment.

2. Experiment

Co, Fe and Ni powders were subjected to the MA process to obtain ternary $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ alloys. The compositions of alloys were chosen on the basis of the phase diagram reported for bulk, melted Co-Fe-Ni alloys in [4]. We concentrated on Co-rich compositions, because the literature reports indicate that the alloys possess soft magnetic properties. Milling was performed in a Fritsch P5 planetary ball mill with a stainless-steel vial and balls. Both MA processes were conducted up to 100 h under an argon atmosphere.

Thermal treatment (TT) of the mechanosynthesized Co-Fe-Ni alloys was performed in two ways: (1) heating from the room temperature up to 993 K in a calorimeter under an argon atmosphere with the rate of 20 K per min

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and (2) isothermal annealing in a furnace at 1173 K for 1 h in vacuum.

Chemical compositions of the samples after MA and TT were verified by X-ray microprobe measurements. Images of powders at different stages of milling process were taken using scanning electron microscope (SEM) .

X-ray diffraction (XRD) measurements were carried out using a Philips PW1830 diffractometer working in a continuous scanning mode with CuK_α radiation. The lattice constants were determined from the shift of the diffraction lines. The Williamson–Hall approach was used for determination of the average grain sizes D and the mean level of internal strains ε [10].

Mössbauer spectra of mechanosynthesized and thermally processed samples were registered at room temperature in transmission geometry using a source of ^{57}Co in a chromium matrix.

The hysteresis loops measurements were performed using a vibrating sample magnetometer at room temperature in a field up to ± 1.6 T. The temperature dependencies of magnetization were registered on a Faraday balance in a magnetic field up to 1.5 T and heating rate up to 4 K per min.

3. Results and discussion

The final products of MA were powder alloys with an average particle sizes ranged of 10 μm. Thermal treatment caused significant increase of particle sizes even up to about 100–200 μm (Fig. 1). Homogeneity and chemical composition of the samples were verified by X-ray microprobe measurements.

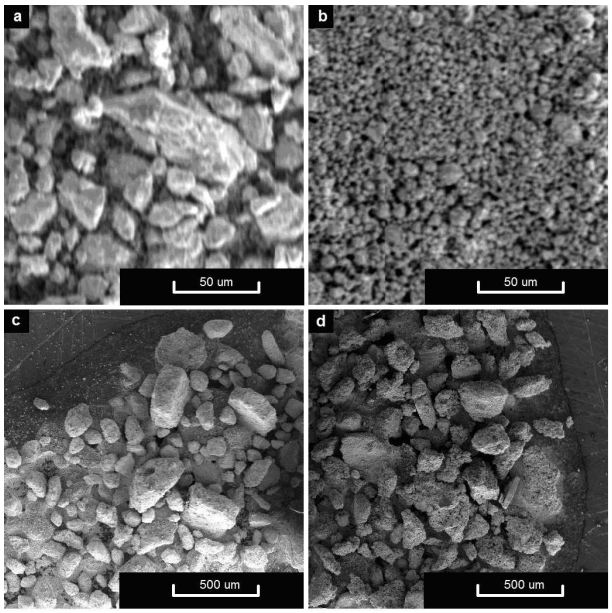

Fig. 1. SEM images of $Co_{50}Fe_{45}Ni_5$ a) after 5 h MA b) after 100 h MA c) after heating d) after annealing.

As XRD studies proved for $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ after mechanical milling disordered solid solutions with bcc crystalline lattice were formed, with lattice constant a equal to 0.2853(1) nm and 0.2851(1) nm respectively. The samples after mechanical alloying were characterized by the relatively high level of internal strains ε, ranged of 1% while TT led to decrease of this value to about 0.2%. It is important to note that powders after MA and TT were in nanocrystalline state with an average grain sizes about tens of nanometers. In the case of $Co_{40}Fe_{50}Ni_{10}$ alloy TT caused formation of a mixture of bcc and small amount of fcc phases. In other cases structures of samples turned out stable. Detailed results of the structural studies are presented in Table I.

TABLE I

Structural data for $Co_{50}Fe_{45}Ni_5$ and $Co_{40}Fe_{50}Ni_{10}$ after MA and heat treatment.

Alloy	State	Lattice	a [nm]	D [nm]	ε [%]
$Co_{50}Fe_{45}Ni_5$	after MA	bcc	0.2851(1)	20(20)	0.80(30)
	heated up to 993 K	bcc	0.2852(1)	100(80)	0.20(5)
	annealed at 1173 K	bcc	0.2851(1)	60(10)	0.15(2)
$Co_{40}Fe_{50}Ni_{10}$	after MA	bcc	0.2853(1)	60(30)	0.95(50)
	heated up	bcc	0.2856(1)	> 100	0.28(5)
	to 993 K	fcc	0.3577(1)	80(80)	0.25(8)
	annealed at 1173 K	bcc	0.2854(1)	> 100	0.20(5)

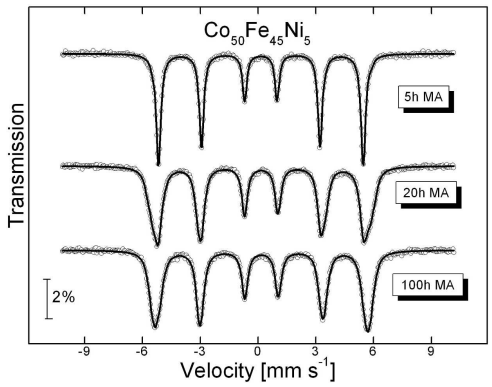

Fig. 2. Mössbauer spectra for $Co_{50}Fe_{45}Ni_5$ alloy registered for different stage of milling process.

Mössbauer spectra registered for $Co_{50}Fe_{40}Ni_5$ after different times of milling process are presented in Fig. 2. For both investigated compositions, the spectra of the samples milled for 5 h were characteristic for α-iron. It may be noted that half-width at half-maximum of the spectral lines increases as the time of milling increases and reach 0.16 mm/s for the final product of alloying. This broadening was the result of decrease of grain sizes, relatively high level of internal strains and alloy formation, i.e. appearing different atomic configurations in the near-

est neighborhood of ^{57}Fe. Taking into account broadening of the lines and the disordered character of alloys hyperfine magnetic field distribution method was used to fit the spectra. Figure 3 as an example presents hyperfine magnetic field (HMF) distributions for mechanosynthesized and thermally treated alloys. The shapes of such distributions were similar for both studied compositions. The average value of HMF, $\langle B_{hf}\rangle$, for $Co_{40}Fe_{50}Ni_{10}$ was by 0.41 T larger than for $Co_{50}Fe_{40}Ni_5$ (Table II) and it was mainly a result of larger contents of iron. Thermal treatment caused decreasing of $\langle B_{hf}\rangle$ value by about 0.2–0.3 T. Only in the case of the heated $Co_{40}Fe_{50}Ni_{10}$ alloy the decrease was grater (i.e. 0.47 T) and was connected with a presence of about 15% of fcc phase in alloy. The quoted 15% was estimated from XRD pattern.

TABLE II

Mössbauer data for $Co_{50}Fe_{45}Ni_5$ and $Co_{40}Fe_{50}Ni_{10}$ alloys. $\langle B_{hf}\rangle$ — average value of HMF, B_{max} — most probable value of HMF, σ_B — disperse of distribution.

Alloy	State	$\langle B_{hf}\rangle$ [T]	B_{max} [T]	σ_B [T]
$Co_{50}Fe_{45}Ni_5$	after MA	33.95	34.31	1.58
	heated up to 993 K	33.75	33.79	0.70
	annealed at 1173 K	33.61	33.79	1.19
$Co_{40}Fe_{50}Ni_{10}$	after MA	34.36	34.31	1.48
	heated up to 993 K	33.89	34.31	1.16
	annealed at 1173 K	34.03	34.31	1.11

TABLE III

Magnetic data for $Co_{50}Fe_{45}Ni_5$ and $Co_{40}Fe_{50}Ni_{10}$ alloys. B_S — saturation magnetization, H_C — coercive field, μ_{eff} — effective magnetic moment per formula unit.

Alloy	State	B_S [T]	H_C [Oe]	μ_{eff} [μ_B]
$Co_{50}Fe_{45}Ni_5$	after MA	2.05	65.7	2.02
	heated up to 993 K	—	—	1.95
	annealed at 1173 K	—	—	2.10
$Co_{40}Fe_{50}Ni_{10}$	after MA	2.03	49.8	2.00
	heated up to 993 K	—	—	2.05
	annealed at 1173 K	—	—	2.04

The results of magnetic measurements for $Co_{40}Fe_{50}Ni_{10}$ are presented in Fig. 4 as an example. Both the series of studied alloys exhibit a ferromagnetic behavior up to relatively high temperatures and only weakly depend on thermal treatment. The magnetization of both the $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ alloys remains a slowly varying function of temperature up to about $T_1 = 650$ K. The abrupt reduction of magnetic ordering is observed above T_1. The effective magnetic moment per formula unit, μ_{eff}, was determined and

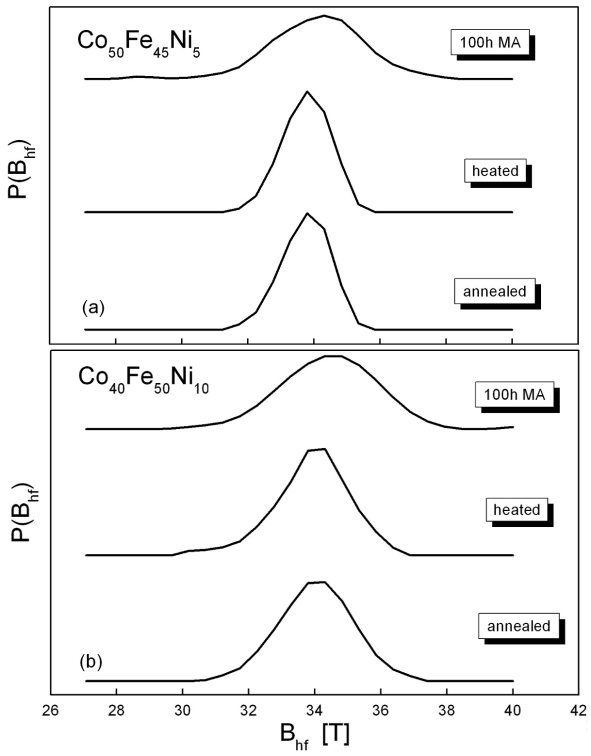

Fig. 3. Hyperfine magnetic field distributions for (a) $Co_{50}Fe_{45}Ni_5$ and (b) $Co_{40}Fe_{50}Ni_{10}$.

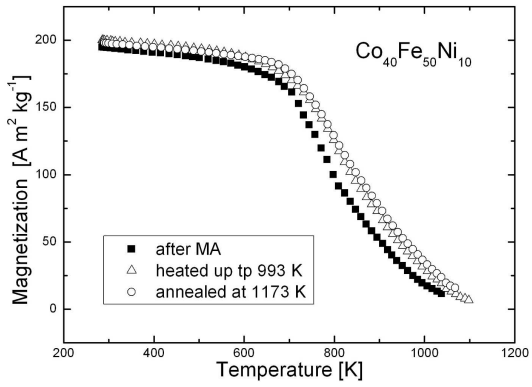

Fig. 4. Dependencies of magnetization on temperature for $Co_{40}Fe_{50}Ni_{10}$ alloy after MA and heat treatment.

listed in Table III. The high temperature part of magnetization curves showed that the Curie temperatures were very close for both alloy series and exceeded 1100 K. The hysteresis loops were measured only for the samples after MA. The values of the saturation magnetization and coercive field are added in Table III.

4. Conclusions

This work follows our systematic investigations concerned on ternary Co-Fe-Ni alloys obtained by mechanical synthesis. Performed studies allowed to state

that prepared by mechanical alloying $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ were homogenous and single phase samples. The structure of the $Co_{40}Fe_{50}Ni_{10}$ alloy was unstable and heating of it caused decomposition into two phases. The final products of MA processes were characterized by relatively broad but smooth and regular HMF distributions similar to Gausian functions what was the result of disordered character of alloys. Thermal treatment in all cases led to a decrease of the level of internal strains and an increase of grain size and as a consequence narrowing of HMF distributions. The values of saturation magnetization and coercive field allowed us to consider $Co_{40}Fe_{50}Ni_{10}$ and $Co_{50}Fe_{45}Ni_5$ alloys as a soft magnetic powders.

Acknowledgments

This work was supported by the grant No. N N202 127836 of the Polish Ministry of Science and Higher Education. The authors want to thank Mr. K. Zabielski for help in measurements using SEM microscope.

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