Rev.Adv.Mater.Sci. 18(2008) 322-325

# THERMAL STABILITY AND HYPERFINE INTERACTIONS OF MECHANICALLY SYNTHESIZED $Co_{40}Fe_{40}Ni_{20}$ ALLOY

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*Received: March 29, 2008*

**Abstract.** Mechanical alloying method was used to obtain $Co_{40}Fe_{40}Ni_{20}$ alloy from the elemental powders. X-ray diffraction studies revealed that the final product of milling was the solid solution with *b.c.c.* lattice and the average grain size of about 36 nm. After heating of the alloy up to 993 K the mixture of two solid solutions with *b.c.c.* and *f.c.c.* lattices with small amount of the third iron-oxide phase was formed. Annealing of the alloy at 1173K during 1 h resulted in the formation of the solid solution with *f.c.c.* lattice. Mössbauer spectroscopy allowed to determine the hyperfine interactions parameters of the obtained phases. Results of the macroscopic magnetic measurements testified that $Co_{40}Fe_{40}Ni_{20}$ alloy had relatively good soft magnetic properties.

## 1. INTRODUCTION

Co-Fe-Ni ternary alloys exhibit good soft magnetic properties [1-3]. We propose the mechanical alloying (MA) method as the potential technology for production of such materials. While the macroscopic magnetic properties of the ternary Co-Fe-Ni alloys obtained by various methods were described in the literature quite well, the hyperfine interactions in these alloys are not well known, especially in the case of the mechanically synthesized alloys and subsequently subjected to thermal treatment.

The aim of this work was: (1) to synthesize $Co_{40}Fe_{40}Ni_{20}$ alloy using MA process and (2) to characterize the structure, hyperfine interactions and macroscopic magnetic properties of the alloy after MA process as well as after heat treatment.

## 2. EXPERIMENT

Co, Fe and Ni elemental powders (purity of 99.9%, initial size of particles 3-10 μm) were subjected to MA process in the high-energy Fritsch P5 planetary ball mill with stainless-steel vial and balls. The milling was performed under an argon atmosphere.

X-ray diffraction (XRD) measurements were carried out using a Philips PW 1830 diffractometer working in a continuous scanning mode with $CuK_\alpha$ radiation. The Williamson-Hall approach was used for determination of the average grain sizes, $D$, and the mean level of internal strains, $\varepsilon$, [4]. Mössbauer spectroscopy (MS) studies were performed at room temperature in standard transmission geometry using a source of $^{57}Co$ in a rhodium matrix. The hysteresis loop was obtained using a vibrating sample magnetometer at room temperature in field

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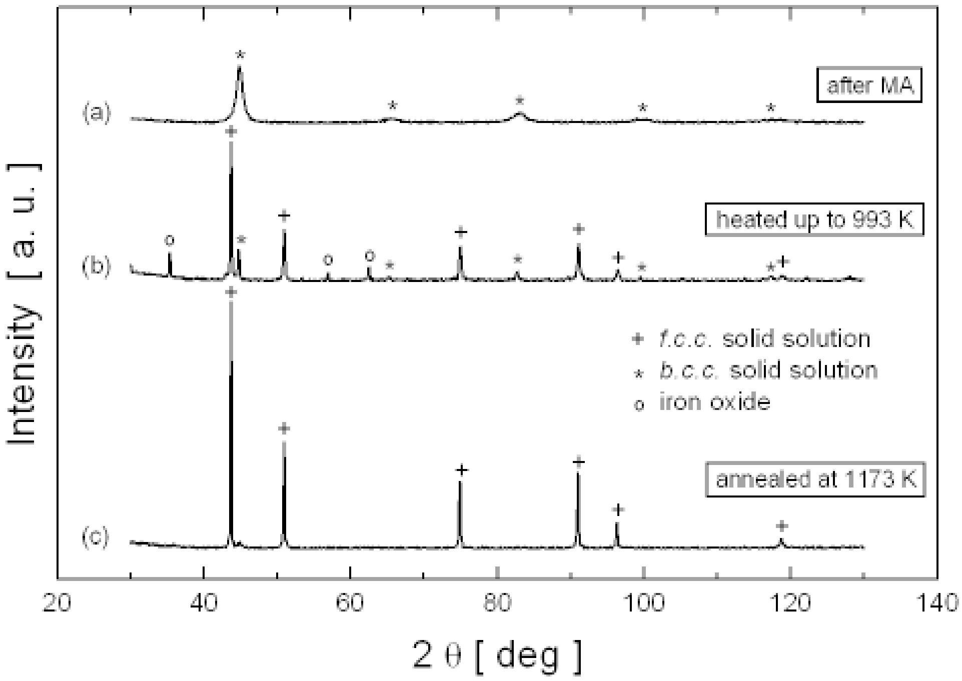


**Fig. 1.** XRD patterns of $Co_{40}Fe_{40}Ni_{20}$ alloy: (a) after MA process, (b) after MA and heating up to 993K, (c) after MA and annealing at 1173K.

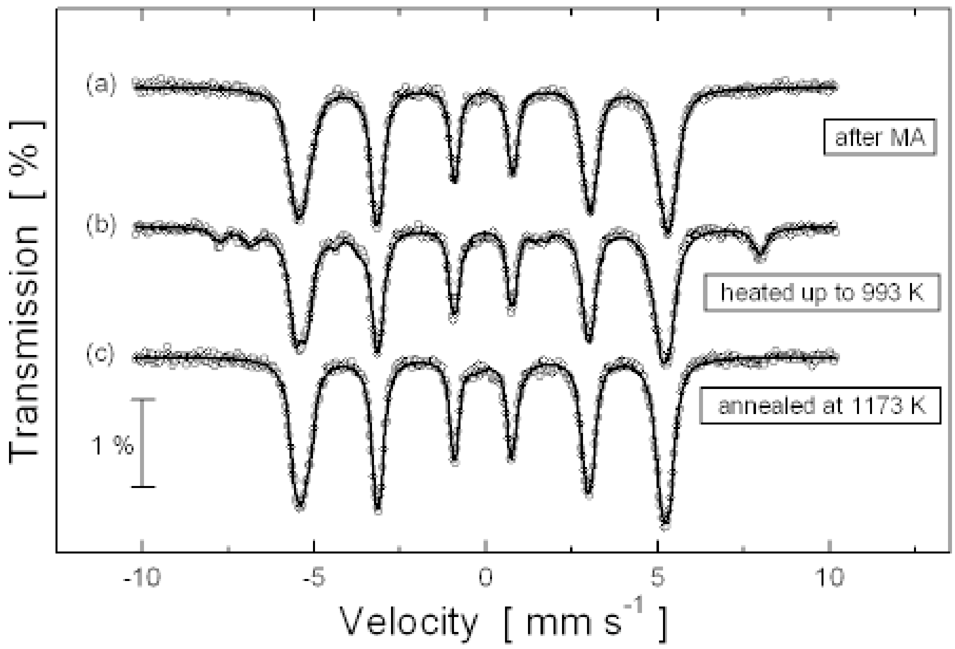


**Fig. 2.** Room-temperature Mössbauer spectra of $Co_{40}Fe_{40}Ni_{20}$ alloy: (a) after MA process, (b) after MA and heating up to 993K, (c) after MA and annealing at 1173K.

up to ±1.6 T. Temperature dependencies of magnetization were measured using a Faraday balance in magnetic field up to 1.5 T.

Thermal treatment of the mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy was performed in two ways: (1) heating from the room temperature up to 993K in a calorimeter under an argon atmosphere with the rate of 20K per min and (2) isothermal annealing in a furnace at 1173K during 1 h.

**Table 1.** Structural data and hyperfine interaction parameters for $Co_{40}Fe_{40}Ni_{20}$ alloy after MA process and after heat treatment; *a* – lattice parameter, *D* – average grain sizes, ε - mean level of internal strains, $B_{hf}$ – hyperfine magnetic field, δ - isomer shift relative to α-iron.

| State | Lattice | *a* [nm] | *D* [nm] | ε [%] | $B_{hf}$ [T] | δ [mm s$^{-1}$] |
|---|---|---|---|---|---|---|
| after MA | *b.c.c.* | 0.2846(2) | 36(1) | 1.20(5) | 33.09* | 0.01(1) |
| heated up | *b.c.c.* | 0.28524(8) | > 40 | 0.03(5) | 33.15(2) | -0.03(1) |
| to 993 K | *f.c.c.* | 0.35760(6) | > 70 | 0.06(5) | 32.91(2) | 0.04(1) |
| annealed at 1173 K | *f.c.c.* | 0.35807(2) | > 60 | – | 32.75* | 0.01(1) |

* Average value of HMF with a standard deviation about 1 T.

## 3. RESULTS AND DISCUSSION

As XRD measurements proved, the alloy started to form after 10 h of milling when the Bragg peaks of Ni and hexagonal fraction of Co disappeared. The XRD pattern of the final product of milling (after 100 h) is presented in Fig. 1a. Detailed analysis of XRD results allowed to state that during MA process of $Co_{40}Fe_{40}Ni_{20}$ system the disordered solid solution with *b.c.c.* lattice was formed. The obtained result does not agree with the phase-diagram for the bulk Co-rich Co-Fe-Ni alloys obtained by melting [3], where the mixture of *b.c.c.* and *f.c.c.* phases occur for the same chemical composition. However, this difference is caused by the preparation method. The $Co_{40}Fe_{40}Ni_{20}$ alloy obtained by melting in an arc furnace was then melted several times to assure homogeneity [3]. In our case, the alloy obtained after MA process is a single-phased, however this state may be non-equilibrium. To check the stability of the structure of the mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy, thermal treatment was performed. XRD patterns of the alloys heated up to 993K and annealed at 1173K are presented in Fig. 1b and c, respectively. It may be seen that in the pattern obtained for the alloy heated up to 993K the diffraction lines from *b.c.c.* and *f.c.c.* phases coexist with the third phase, which origin will be explained later. Annealing of the mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy at elevated temperature, 1173K, resulted in the formation of the solid solution with *f.c.c.* lattice. All the structural parameters for the $Co_{40}Fe_{40}Ni_{20}$ alloy after MA process as well as after heat treatment are listed in Table 1.

Mössbauer spectroscopy confirmed XRD results. The MS spectrum for the final product of MA process is presented in Fig. 2a. The fitting of the spectrum using the hyperfine magnetic field, (HMF), distribution proved that in the mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy $^{57}Fe$ atoms have many different surroundings by Co, Fe and Ni atoms. The superposition of these configurations gives the average value of the HMF of about 33.09 T.

Mössbauer spectroscopy allowed also to recognize the phases formed during thermal treatment of $Co_{40}Fe_{40}Ni_{20}$ alloy. The suitable Mössbauer spectra are presented in Figs. 2b and 2c. The best numerical fitting of the spectra was achieved when four components were used for the alloy heated up to 993K and the HMF distribution for the alloy annealed at 1173K. The hyperfine interaction parameters obtained from the fitting are listed in Table 1. In the spectrum of the heated alloy besides two main components that were attributed to the *b.c.c.* and *f.c.c.* phases, additional two sextets with high values of the HMF (49 and 46 T) were observed (Fig. 2b). These sextets have the parameters characteristic for $Fe_3O_4$ [5].

Macroscopic magnetic measurements were performed for mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy. From the dependencies of magnetization on the temperature the effective magnetic moment per formula unit, $\mu_{eff}$, and the Curie temperature, $T_C$, were determined. The value of $\mu_{eff}$ raised monotonically from 1.47 to 1.84 $\mu_B$ with the milling time varying from 1 to 100 h. The $T_C$ was equal to 1090K for the final alloy. From the hysteresis loop measurement the values of the saturation magnetization, $B_s$ = 175 A m$^2$ kg$^{-1}$, and the coercive field, $H_c$ = 23.7 Oe, were obtained. The $B_s$ and $H_c$ values of the same order of magnitude were observed for soft magnetic Co-Fe-Ni thin films [3].

## 4. CONCLUSIONS

The MA process seems to be a promising method for production of soft magnetic Co-Fe-Ni powders. The $Co_{40}Fe_{40}Ni_{20}$ alloy after MA process is in non-equilibrium state. The phase composition after heat treatment of the alloy depends on the heating conditions. The XRD analysis proved that the mechanosynthesized $Co_{40}Fe_{40}Ni_{20}$ alloy had a nanocrystalline structure with the relatively high level of internal strains. In the alloys subjected to thermal treatment the average grain sizes were significantly higher than in the alloy after MA process and the suitable phases had practically non-damaged lattices. The contamination of the powders with an iron-oxide can have occurred during the powder handling.

The values of the saturation magnetization and the coercive field allowed to consider that mechanically synthesized $Co_{40}Fe_{40}Ni_{20}$ alloy had relatively good soft magnetic properties.

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