Journal of Magnetism and Magnetic Materials 324 (2012) 2501–2505

Contents lists available at SciVerse ScienceDirect

Journal of Magnetism and Magnetic Materials

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

Study of magnetic phases in mechanically alloyed $Fe_{50}Zn_{50}$ powder

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ARTICLE INFO

Article history:
Received 21 October 2011
Received in revised form
7 February 2012
Available online 26 March 2012

Keywords:
Nanostructured material
Mechanical alloying
Magnetic measurement
Mössbauer spectroscopy

ABSTRACT

The mechanosynthesis of $Fe_{50}Zn_{50}$ alloy resulted in the formation of the bcc Fe(Zn) solid solution after 20 h of milling. Structural transformations induced by mechanical alloying and heating, and magnetic properties of the powders were studied by Mössbauer spectroscopy, X-ray diffraction, Faraday balance and vibrating sample magnetometry techniques. All alloys studied exhibit strong magnetic ordering with Curie temperatures close to 900 K. Room temperature Mössbauer measurements revealed distinguished magnetic environments in the samples. The decrease of coercivity with prolonged milling time was attributed to the reduction or averaging of local magnetic anisotropies.

1. Introduction

Due to the proper mechanical properties and high atmospheric corrosion resistance the Fe–Zn system is widely used in the industry to produce protective coatings for steel surfaces [1]. However, large differences in melting temperatures and diffusivities of Fe and Zn hamper traditional melting technologies. The majority of previous research on the Fe–Zn system has been devoted to non-magnetic Zn-rich intermetallic phases. On the other hand, ferromagnetic Fe–Zn alloys with Zn content up to about 60 at% and with Curie temperatures exceeding room temperature were obtained by the vapor quenching technique [2]. Recently, it was confirmed that the ball milling technique operating at relatively low temperatures allows one to overcome these limitations and to produce the $Fe_{80}Zn_{20}$ alloy [3]. This initial result along with similar studies [4] encourages investigation on which other magnetic Fe–Zn alloys could be synthesized by ball milling.

Our previous study [3], which was focused on the preparation of $Fe_{80}Zn_{20}$ alloys under various milling conditions, revealed a strong correlation between the preparation method and the final phase composition. The present work is aimed at deeper structural and magnetic investigation of the phases contained in the $Fe_{50}Zn_{50}$ alloy prepared by mechanosynthesis at various milling times and heated up to 900 K. The scientific interest in this alloy is additionally justified by a very limited magnetic characterization of the Fe–Zn system available in literature. In the present study, complementary methods were used to characterize the phases formed due to ball milling and heating of $Fe_{50}Zn_{50}$ alloy. The structure of the powders was studied by a combination of Mössbauer spectroscopy and X-ray diffraction methods. ^{57}Fe Mössbauer spectroscopy, which is very sensitive to iron atomic surroundings, has been successfully applied in the studies of iron–zinc alloys and intermetallics obtained by various preparation techniques [2,5–7]. In the present work, Mössbauer measurements provided information regarding local chemical and structural environments around Fe nuclei at each step of the mechanosynthesis. Analysis of Mössbauer spectra allowed the identification of iron-containing phases as well as the quantitative analysis of their relative fractions. Characterization of magnetic properties of the $Fe_{50}Zn_{50}$ samples was performed on the basis of the data obtained from magnetization, hysteresis loop and Mössbauer measurements.

2. Experimental details

The elemental powders of Fe and Zn with a purity of at least 99.8% and particles sizes less than 50 μm were used in the mechanical alloying process. The $Fe_{50}Zn_{50}$ mixture (at%) was subjected to high-energy ball milling. A Fritsch Pulverisette P5 planetary ball mill, equipped with a hardened steel vial and balls (10 mm in diameter), was employed for milling with a rotational speed of 250 rpm. The mass of the milled powders was 10 g and ball-to-powder weight ratio was 10:1. The milling experiments and all powder handling were performed under protective atmosphere of argon. Small amounts of powders were withdrawn after selected milling times for structural examinations. The structure

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http://dx.doi.org/10.1016/j.jmmm.2012.03.022

of the powders at different stages of milling and after completion of the process, as well as after annealing, was investigated by the X-ray diffraction (XRD) method in a Rigaku MiniFlex II diffractometer using CuK_α radiation ($\lambda = 0.15418$ nm).

The Mössbauer measurements of all powder samples were performed in transmission geometry at room temperature. A constant-acceleration spectrometer with a 25 mCi ^{57}Co-in-Rh source was used. The spectra were fitted assuming a Lorentzian profile of lines using the NORMOS program. The relative fractions of the spectral components related to the identified phases were calculated as a ratio of the area of the relevant subspectrum to the total spectral area, assuming similar Debye–Waller factors for each phase. Isomer shifts are referred to α-Fe standard. Temperature variation of magnetization was measured by means of a Faraday balance up to 1000 K in a magnetic field of 1.5 T. Magnetic hysteresis loops were recorded with a vibrating sample magnetometer during a full cycle in a magnetic field with an amplitude of 1600 Oe. Temperature was regulated with 0.1 K accuracy. The magnetic field was stabilized when measuring hysteresis loops point by point.

3. Results

3.1. XRD results

Fig. 1 shows a sequence of XRD patterns registered for the samples subjected to mechanical alloying for increasing milling times. It can be seen that the diffraction lines belonging to Fe and Zn gradually disappear and new lines become visible after 20 h of processing. The new diffraction lines are significantly shifted to smaller angles as compared with the starting bcc Fe lines. Therefore, we attribute these lines to the formed bcc Fe(Zn) solid solution. The calculated lattice parameter of 0.2965 ± 0.0005 nm is significantly larger than that of pure bcc Fe phase (0.287 nm). Average crystallite sizes of the bcc Fe(Zn) solid solution are about 10 ± 2 nm as estimated by the Scherrer method. Traces of the bcc Fe phase at 44.5° can be also observed.

Fig. 2 shows the XRD pattern recorded for the sample heat-treated after 20 h of mechanical alloying (heating up to 900 K). The diffraction lines can be assigned to two phases: bcc Fe (squares) and Fe_4Zn_9 (Γ phase; triangles). The lines attributed to Fe are shifted slightly, which indicates that the bcc lattice contains some zinc. The phase composition of the alloy after heat treatment seems to be close to the equilibrium one. One may notice that during heating, the average crystallite sizes raise to 30 and 40 nm for the bcc Fe(Zn) and Fe_4Zn_9 phases, respectively. All XRD patterns do not show any traces of the ferrite materials, which shows that the samples were prepared in a clean argon atmosphere that was free of oxygen [3].

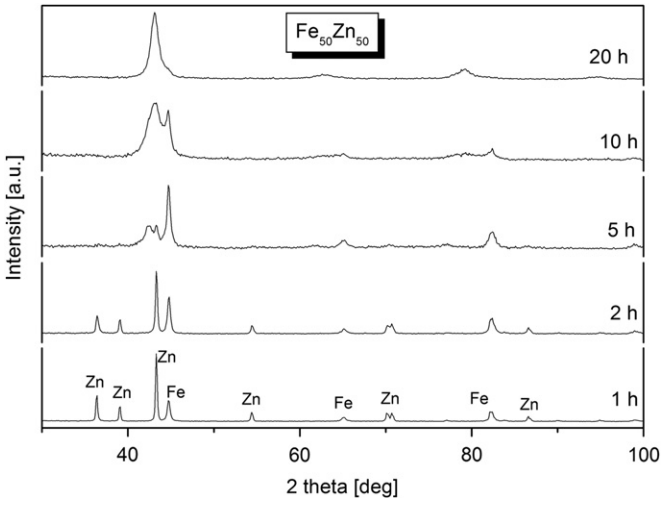

Fig. 1. XRD patterns registered for the samples subjected to mechanical alloying for increasing periods.

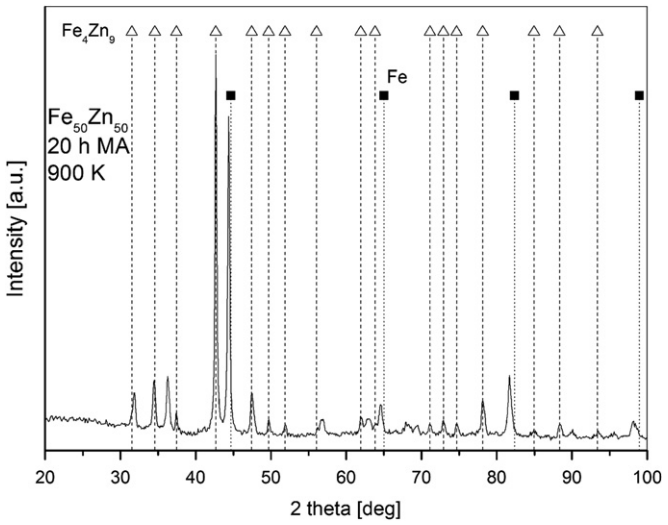

Fig. 2. XRD pattern for the sample heated up to 900 K after 20 h of mechanical alloying. Squares and triangles correspond to the bcc Fe and Fe_4Zn_9 phases, respectively.

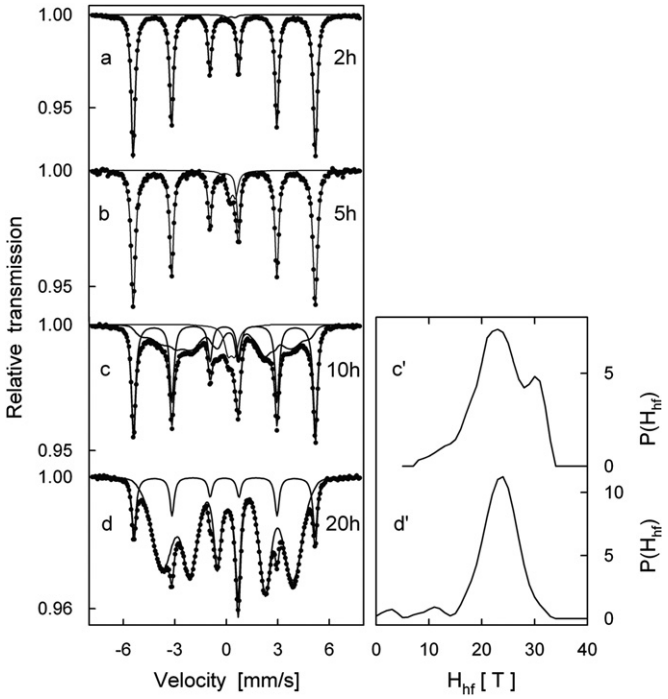

Fig. 3. Mössbauer spectra of alloys milled for 2–20 h (a–d) and the corresponding hyperfine field distributions (c′, d′).

3.2. Mössbauer spectra of alloys milled up to 20 h

^{57}Fe Mössbauer spectroscopy measurements allowed us to study changes of local atomic environments around Fe nuclei induced by the milling process. The spectra recorded as a function of the milling time are shown in Fig. 3. They were fitted with two or three spectral components. The hyperfine parameters obtained