Mössbauer Study of Some Intermetallic Compounds Fe$_{80-x}$Ni$_x$B$_{20}$ (x = 8, 16, 24, 28)

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Fe-based nanocrystalline alloys were prepared by the melt-spinning technique and characterized by X-ray diffraction, magnetostatic and Mössbauer effect methods. The Mössbauer spectroscopy allows to study the local environment of the Fe atoms in the investigated Fe$_{80-x}$Ni$_x$B$_{20}$ (x = 8, 16, 24, 28) compounds and showing the changes in the structure due to the changing of Ni addition. Combination of X-ray diffraction and Mössbauer spectroscopy results confirm formation of different number of phase complex, including the α-FeNi, γ-FeNi, FeB, Fe$_2$B, Fe$_3$B in the investigated materials together with increase of Ni content.

1. Introduction

Nanocrystalline alloys rich in Fe prepared from amorphous precursors by crystallization process represent an important part of soft magnetic materials. Especially, Fe–Ni alloys around the Invar composition of 35 at.% Ni are known to show various anomalies in physical properties. These compounds, based on iron and nickel, are in the constant focus of interest in physical and material research as well as in industrial applications, as they represent the class of construction materials and show unique magnetic properties. For example, two magnetic moments of the Fe atoms exist in fcc structure of Fe–Ni alloys in the Invar region: high moment with high volume and small or zero moment with small volume. These di?erent magnetic moments are result of local environment and can influence on observed a very small thermal expansion coefficient near room temperature for these alloys. Alloys containing Fe and Ni together with further additions of specific elements are attractive from mechanical, magnetic and structural point of view [1, 2].

In the last years, alloys with crystallite sizes less than 100 nm have attracted attention due to some of their magnetic properties such as effective permeability and saturation magnetic flux density [3, 4]. The Invar FeNi alloys were prepared mostly by arc-melting. The obtained compounds are polycrystalline and their shape is difficult to use in potential applications. We want to apply melt-spinning technique to obtain alloys which are cast as ribbon shaped metallic glasses with thickness range from 0.05 mm to 0.2 mm. We are also looking for a nickel concentration for which the content of γ-FeNi

phase rich in iron will be the highest because then we expect the occurrence of Invar effects.

2. Experimental procedure

The studies were performed on Fe$_{80-x}$Ni$_x$B$_{20}$ (x = 8, 16, 24, 28) nanocrystalline compounds prepared by the chill-block melt-spinning technique (described in [5]).

The XRD measurements were performed at room temperature using the Siemens D5000 X-ray diffractometer with Cu K$_\alpha$ radiation. From these measurements the type of crystal structure was determined and also the average crystallites size D from X-ray line broadening were calculated using the Scherrer equation.

The magnetic measurements of the investigated samples were performed using magnetic balance in the temperature range of 300–1100 K.

The Fe^{57} Mössbauer spectra were recorded at room temperature using a constant acceleration spectrometer with Co^{57}:Rh source. Metallic iron foil was used for velocity calibrations of the Mössbauer spectrometer.

3. Result and discussion

Four samples with various nickel content were compared using room temperature Mössbauer spectroscopy and X-ray analysis. Table I summarizes the detected crystal phases, average crystalline sizes of all the phases recognized in XRD patterns and an estimation of a percentage contribution. For illustration, X-ray diffraction patterns and Mössbauer spectra of the investigated materials are shown together with their decomposition on Figure 1 and 2, respectively. The Mössbauer hyperfine interactions parameters of fitted components are in agreement with those presented in literature [5–7]. Small differences in values of hyperfine interactions parameters can arise from substitution of iron atoms by nickel atoms.

All the studied compounds were nanocrystalline and the average crystalline sizes are ranged from 43 nm to

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51 nm. Together with increase of nickel content the crystalline sizes decrease what can suggested that high nickel concentration leads to increase probability of crystallization of compounds. The substitution of small amounts of Ni ($x = 8$, 16) for Fe in $Fe_{80-x}Ni_xB_{20}$ compounds generally results in change of structure of nanograins FeNi from body-centered cubic (α) to face-centered cubic (γ). The analysis of phase evolution with of Ni content indicates that an increase of nickel concentration leads to the formation of different ratio of nanocrystalline phases (Table I) and consequently to a change of magnetic properties. Together with increase of Ni content in investigated compounds content of soft Fe_3B and Fe_2B phases increases content, building the well known exchange coupled nanocomposite structures. If iron atoms build mainly these two phases this proves that nickel is built in majority in FeB and FeNi structures.
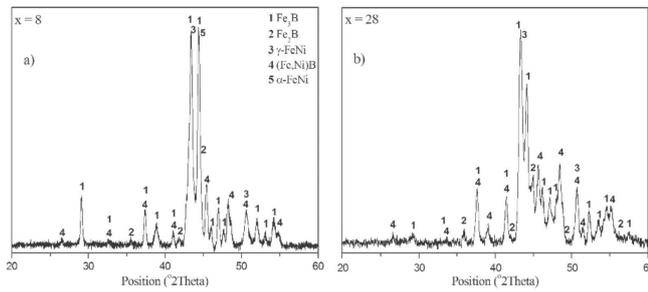
Fig. 1. X-ray diffraction patterns of the Ni-low a) and Ni-rich b) $Fe_{80-x}Ni_xB_{20}$ samples.
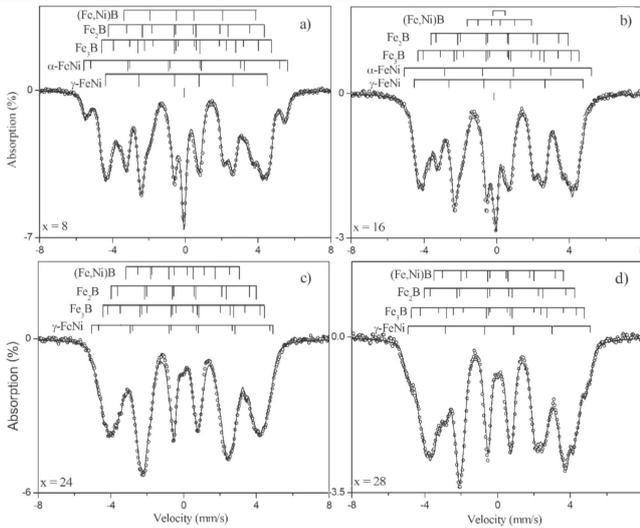
Fig. 2. Mössbauer phases analysis of $Fe_{80-x}Ni_xB_{20}$ (a–d) compounds for $x = 8$, 16, 24, 28 (a–d).

Mössbauer spectrum of $Fe_{80-x}Ni_xB_{20}$ ($x = 8$) was fitted using ten components. Three sextets with hyperfine magnetic fields $H = 28.9$ T, 25.0 T, and 22.6 T are connected with Fe_3B phase, two sextets with $H = 26.3$ T and 20.4 T with Fe_2B phase and sextet with $H = 22.3$ T

Crystal phases, percentage contribution P of a given phase (obtained from the Mössbauer spectra) and average crystalline sizes D of all the phases recognized in XRD patterns for the studied compounds.

Compound	D [nm]	Phases	P [%]
$Fe_{72}Ni_8B_{20}$	51	Fe_3B, Fe_2B, (Fe,Ni)B, γ-FeNi, α-FeNi	28, 23, 20, 20, 9
$Fe_{64}Ni_{16}B_{20}$	47	Fe_3B, Fe_2B, (Fe,Ni)B, γ-FeNi, α-FeNi	46, 26, 12, 13, 3
$Fe_{56}Ni_{24}B_{20}$	47	Fe_3B, Fe_2B, (Fe,Ni)B, γ-FeNi	37, 27, 25, 11
$Fe_{52}Ni_{28}B_{20}$	43	Fe_3B, Fe_2B, (Fe,Ni)B, γ-FeNi	36, 40, 19, 5

is connected with (Fe,Ni)B phase. The α-FeNi phase is represented by H=34.4 T and 32.8 T. Sextet with $H = 27.6$ T and single line with isomer shift value –0.07 mm/s belong to the γ-FeNi phase.

Sextets with $H = 27.3$ T, 25.6 T, 20.3 T on the Mössbauer spectrum of $Fe_{80-x}Ni_xB_{20}$ ($x = 16$) are connected with the Fe_3B phase and sextets with $H = 23.7$ T and 21.7 T with Fe_2B phase. In this compound also exists γ-FeNi phase visible on the Mössbauer spectrum as a sextet with hyperfine magnetic field 28.9 T and singlet with isomer shift –0.02 mm/s. Small part of this Mössbauer spectrum takes sextet of α-FeNi with $H = 31.6$ T. Doublet with isomer shift 0.11 mm/s and quadrupole splitting 0.59 mm/s and sextet with $H = 10.9$ T belong to (Fe,Ni)B phase.

Mössbauer spectrum of $Fe_{80-x}Ni_xB_{20}$ ($x = 24$) was fitted using nine components. With Fe_3B phase are related sextets where $H = 27.6$ T, 23.1 T, 21.4 T and with Fe_2B phase H $= 26.0$ T and 24.5 T. Four other sextets with $H = 31.0$ T, 29.4 T and $H = 19.4$ T, 15.0 T are connected with γ-FeNi and (Fe,Ni)B, respectively.

On the Mössbauer spectrum of $Fe_{80-x}Ni_xB_{20}$ ($x = 28$) are visible nine components. As in the case of other spectra three hyperfine magnetic fields 27.2 T, 25.4 T and 21.7 T belong to Fe_3B and two with 23.6 T and 21.4 T to Fe_2B phase. The γ-FeNi phase is represented by $H = 31.6$ T and 29.7 T and the (Fe,Ni)B phase by $H = 19.4$ T.

Figure 3 presents thermomagnetic curves M(T) in the temperature range from 300 K to 1100 K for all the studied $Fe_{80-x}Ni_xB_{20}$ ($x = 8$, 16, 24, 28) alloys. Such curves indicates on existence different crystal phases and confirm X-ray and Mössbauer spectroscopy results. The increase of magnetization observed at temperature about 710 K for alloys with $x = 8$ and $x = 16$ is surely related to structural transformation of α-FeNi to γ-FeNi phase [6]. As can be seen, with increasing of nickel concentration in the investigated compounds temperatures of magnetic transitions characteristic for different crystal phases shift towards to lower temperatures. It may be result of substitution of nickel for iron atoms in existing nanocrystalline phases.
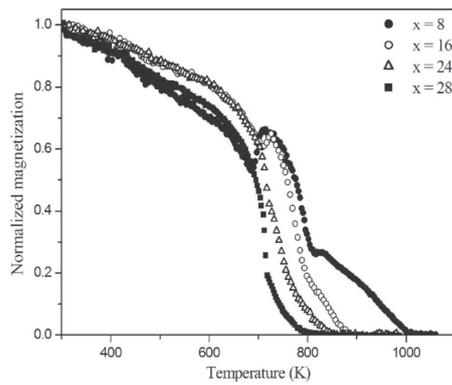
Fig. 3. Thermomagnetic curves M(T) for all the studied compounds.

4. Conclusions

The main conclusions of the present paper, in relation to the $Fe_{80-x}Ni_xB_{20}$ (x = 8, 16, 24, 28) nanocrystalline alloys, can be summarized as follows:

- high nickel concentration (x > 8) lead to increase probability of crystallization of compounds;

- for low nickel concentration (x = 8) the content of γ-FeNi phase rich in iron was the highest;

- increase of nickel concentration leads to the formation of different ratio of nanocrystalline phases and changing magnetic properties of these phases;

- existence of structural transformation of α-FeNi to γ-FeNi phase at temperature 710 K was observed.

References

[1] G. Vlasak, P. Svec, M. Kuzminski, A. Slawska-Waniewska, B. Butvinova, P. Butvin, J. Hosko, H. Bluhm, *J. Alloy Comp.* **509**, S64 (2011).

[2] M. Arshed, M. Siddique, M. Anwar-ul-Islam, A. Ashfaq, A. Shamim, N.M. Butt, *Solid State Common.* **98**, 427 (1996).

[3] J.S. Garitaonandia, P. Gorria, L. F. Barquín, J. M. Barandiarán, *Phys. Rev. B* **61**, 6150 (2000).

[4] J. Sitek, J. Degmová, K. Sedlačková, J. Dekan, *J. Modern Phys.* **3**, 274 (2012).

[5] R. Babilas, M. Kądziołka-Gaweł, *Acta Phys. Pol. A* **127**, 573 (2015).

[6] M. Kądziołka-Gaweł, W. Zarek, E. Popiel, *J. Phys.* **217**, 012088 (2010).

[7] M. Mao, Ch. Yang, Z. Cheng, Y. Zhang, B. Shen, L. Yang, F. Li, *J. Phys. Condens. Matter* **4**, 9147 (1997).