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The Crystal and Magnetic Properties of Some Fe–Nb–B–Ni Alloys

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The crystal and magnetic properties of $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) bulk alloys prepared by making use of mould casting technique have been studied by X-ray diffraction, magnetostatic and Mössbauer effect methods. Structural and magnetic properties of investigated bulk alloys have been compared with polycrystalline $Fe_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) alloys. The measurements have shown that the crystal and magnetic nonhomogeneity for bulk alloys are higher than in polycrystalline compounds, which confirms many magnetic wide magnetic hyperfine field distribution. The mean diameters of crystallites for $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ alloys was calculated from X-ray line broadening and were bigger than 10 nm. The mean magnetic moment and average magnetic hyperfine fields decrease with increase of nickel concentration in investigated compounds.

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1. Introduction

During the last decade a considerable number of bulk and nanocrystalline materials have been intensively studied due to the promising properties to be used for soft magnetic applications [1, 2]. These materials are obtained by different techniques, including mechanical alloying or reactive melting. The samples usually have a particle the size of several μm, but they consist of nanometric crystallites. Iron based compounds are probably the most relevant from a commercial point of view, not only due to its iron natural abundance, but also because of its low cost and excellent conjunction of magnetic properties like large saturation magnetization, low coercive field and high permeability they characterized high mechanical strength and good resistance to corrosion [3].

In the present work we have reported the influence of Ni concentration on structural and magnetic properties of bulk $(Fe_{80}Nb_4B_{16})_{1-x}Ni_x$ ($x = 0.1$, 0.2, 0.4) alloys series. The structural and magnetic properties of investigated bulk alloys have been compared with polycrystalline $Fe_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) alloys. These properties have been studied by X-ray diffraction, magnetostatic and Mössbauer effect methods.

2. Experimental procedure

The samples $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) were prepared by means of the mould casting technique. The sample technology consists of two steps. Firstly, a $Fe_{80}Nb_6B_{14}$ amorphous melt spun ribbon was melted with a proper amount of Ni (purity of 99.96%) by the use of typical arc furnace in an inert gas atmosphere. In the second step the obtained alloy ingots were casting in a cooper mould using the self-designed vacuum suction apparatus [4]. The applied mould allows obtaining the bulk rods of 1.5 mm in diameter and about 3 cm in length. The polycrystalline $Fe_{1-x}Ni_x$ alloys were prepared by arc-melting stoichiometric quantities of the starting metals in an argon atmosphere.

The XRD measurements were performed at room temperature using the Siemens D5000 X-ray diffractometer and Cu K_α 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 Fe–Ni samples were performed using the Faraday method in temperature range 300–700 K and in magnetic fields up to 1.2 T and for bulk alloys SQUID magnetometer was used in temperature up 1100 K and magnetic fields up to 7 T.

The Fe^{57} Mössbauer spectra were recorded in a room temperature using a constant acceleration spectrometer with Co^{57}:Cr source. Metallic iron powder was used for velocity calibrations of the Mössbauer spectrometer. All spectra were fitted by means of a hyperfine field distribution using the Hesse–Rübartsch procedure [5] with a linear correlation between isomer shift and hyperfine magnetic field.

3. Result and discussion

The XRD diffraction pattern for investigated bulk $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) alloys were broad, which can be the result of the composition fluctuation due to the presence of regions with different iron concentration. Figure 1 shows the XRD patterns for selected compounds.

The X-ray diffraction measurements showed that $Fe_{1-x}Ni_x$ alloys exhibited cubic phases bcc ($x = 0.1$, 0.2) and fcc ($x = 0.4$). The XRD diffraction patterns for all bulk samples additionally have been shown Fe_3B, Fe–B phases and small presence of Nb with small addition of Fe. It is difficult to say which phase in bulk alloys is responsible to the some peaks on the XRD pattern because they are weak and broad. In order to make the phases determination more precise are the Mössbauer measurements. The average crystallite sizes were calculated from X-ray line broadening of main peak and

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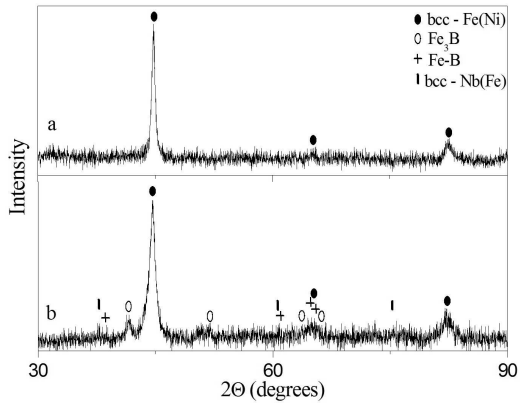

Fig. 1. The XRD pattern obtained for $Fe_{0.8}Ni_{0.2}$ alloy (a) and for $(Fe_{80}Nb_6B_{14})_{0.8}Ni_{0.2}$ bulk alloy (b).

were in the range from about 12 nm to about 14 nm for $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ bulk alloys and from 29 nm to 33 nm for $Fe_{1-x}Ni_x$ alloys, Table. However, in the bulk alloys Fe–Ni nanocrystallites phases are dominating.

TABLE

Crystal structure and phases contribution obtained from the Mössbauer spectra, the Curie temperature T_c, magnetic moment per atom μ, average hyperfine magnetic fields B_{hf} at room and mean diameter of the main phase D temperature for the investigated compounds.

Compound	Phases and contribution [%]	T_c [K]	μ [μ_B/at.]	D [nm]	B_{hf} [T]
$(Fe_{80}Nb_6B_{14})_{0.9}Ni_{0.1}$	Fe-Ni (bcc) 51%, Fe_3B 27%, Fe-B 15%, Nb(Fe) (bcc) 7%	560, 942	1.57	12	25.1
$Fe_{0.9}Ni_{0.1}$	bcc 100	>700	2.41	33	33.7
$(Fe_{80}Nb_6B_{14})_{0.8}Ni_{0.2}$	Fe-Ni (bcc) 44%, Fe_3B 18%, Fe-B 30%, Nb(Fe) (bcc) 8%	650, 917	1.44	12	24.5
$Fe_{0.8}Ni_{0.2}$	Fe-Ni (bcc 55%/fcc 5%)	>700	2.35	29	31.1
$(Fe_{80}Nb_6B_{14})_{0.6}Ni_{0.4}$	Fe-Ni (bcc) 23%, Fe_3B 32%, Fe-B 30%, Nb(Fe) (bcc) 5%	510, 875	1.35	14	23.3
$Fe_{0.6}Ni_{0.4}$	fcc 100%	515	1.74	29	27.3

Magnetic studies show that the investigated compounds are ferromagnetic. The temperature variations of magnetization $M(T)$ for selected investigated compounds are presented in Fig. 2 and values of temperatures T_c connected with magnetic transformation determined from $M^2(T)$ curves are listed in Table. The shape of magnetization curve versus temperature indicates on existence different crystal phases in bulk alloys. The visible increase of magnetization with increase of temperature is surely related to crystallization of the some residual amorphous phase. Figure 3 shows magnetic isotherms at room temperature for all investigated compounds. The bulk alloys achieve the magnetic saturation at much lower magnetic fields than the polycrystalline Fe–Ni compounds.

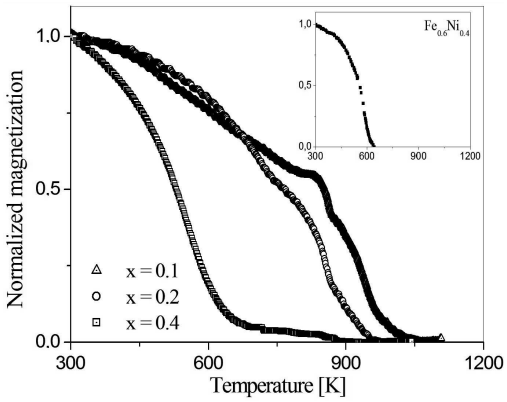

Fig. 2. The normalized thermomagnetic curves $M(T)$ for $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ bulk alloys and for selected $Fe_{0.60}Ni_{0.40}$ alloy (inset).

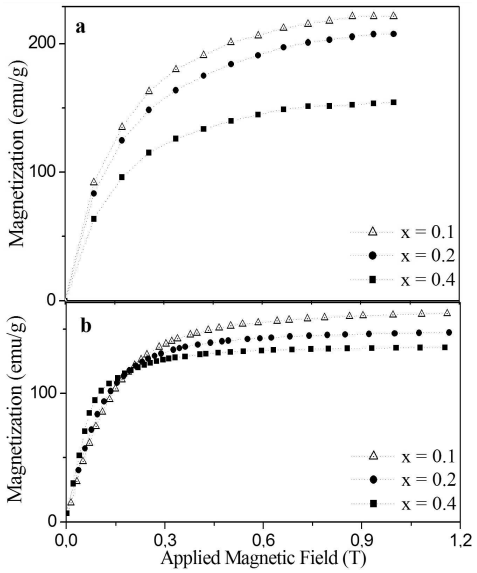

Fig. 3. Room temperature Mössbauer spectra of $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ and $Fe_{1-x}Ni_x$ compounds for $x = 0.1$, 0.2 and 0.4.

This allows to suppose that the $(Fe_{80}Nb_4B_{16})_{1-x}Ni_x$ compounds possess a negligible magnetic anisotropy. The $Fe_{1-x}Ni_x$ alloys saturated in magnetic fields higher than 0.9 T. A precise analysis of the magnetic isotherms showed that the approach to magnetic saturation is mainly determined by magnetic inhomogeneities connected with the atomic disorder of the crystal structure. The values of magnetic moments per atom μ estimated from the magnetic isotherms at 300 K are listed in Table. The magnetic moments connected with bulk alloys are considerably smaller than the magnetic moments of Fe–Ni alloys. Smaller values of μ of bulk compounds in comparison with polycrystalline alloys arise from smaller value of iron concentration.

Room temperature Mössbauer spectra and diagrams of the hyperfine magnetic field B_{hf} distribution (HMF) of $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ and $Fe_{1-x}Ni_x$ ($x = 0.1$, 0.2 and 0.4) alloys are shown in Fig. 4 and Fig. 5, respectively.

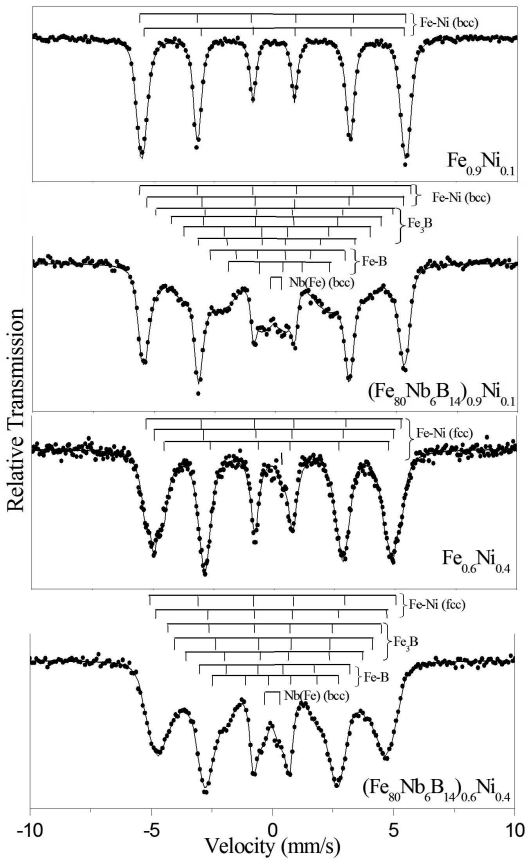

Fig. 4. Room temperature Mössbauer spectra of investigated $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ and $Fe_{1-x}Ni_x$, alloys for $x = 0.1$ and 0.4.

The Mössbauer studies of the investigated alloys confirm the results of structural and magnetic measurements. From these measurements precisely determination of crystal phases is possible, Table. The bcc phase in the studied alloys can be seen as a narrow peak concentrated at the HMF at about 33.5 T ($x = 0.1,\ 0.2$). The fcc phase appears on hyperfine magnetic fields distributions as a peak concentrated near fields 30 T ($x = 0.4$). The hyperfine fields distributions of bulk alloys for fields smaller than 30 T are broad and connected with iron–boron phases [6]. The statistical location of boron atoms in the crystal lattice leads to an increasing number of different crystallographic and magnetic positions of Fe atoms. The bcc niobium phase is manifested themselves on HMF distributions as fields smaller than 6 T. The fitted values of the average HMF are listed in Table. On the hyperfine magnetic fields distributions of all investigated compounds are visible B_{hf} with values higher than for pure ferromagnetic iron (33 T). These values are result of local environment of Fe atoms. If iron atom in bcc crystal structure has from one to about three nickel atoms in nearest neighborhood then magnetic moment on Fe atom increases up to value about 2.4 μ_{B} [7]. The Fe atom has high magnetic moment ($\approx$2.8 μ_{B}) when it is surrounded from zero to about nine Fe atoms in fcc crystal structure [8]. The mean hyperfine magnetic fields

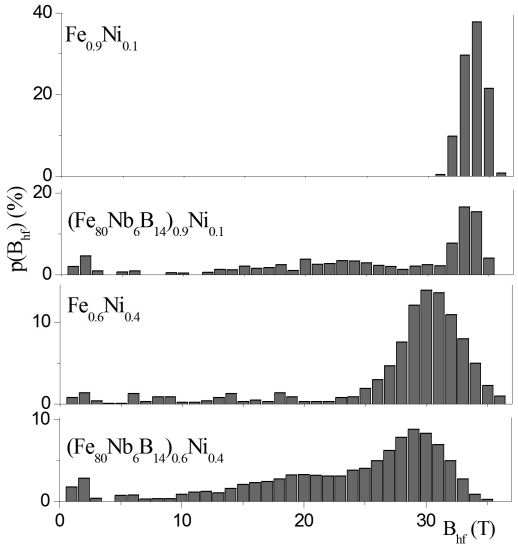

Fig. 5. Hyperfine magnetic field B_{hf} distributions of investigated $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ and $Fe_{1-x}Ni_x$, alloys for $x = 0.1$ and 0.4.

of $(Fe_{80}Nb_6B_{14})_{1-x}Ni_x$ alloys are smaller than fields of $Fe_{1-x}Ni_x$ alloys. It is probably connected with a smaller concentration of Fe atoms. Also, the Fe–Ni nanocrystallites with high magnetic moment are surrounded by Fe–B regions which also leads to devaluation of average hyperfine magnetic fields B_{hf} and magnetic moments μ of bulk alloys.

4. Conclusions

It has been shown that $(Fe_{80}Nb_4B_{16})_{1-x}Ni_x$ ($x = 0.1$, 0.2, 0.4) bulk alloys obtained by means of the mould casting technique have a crystal size more than three times smaller than crystallites in polycrystalline $Fe_{1-x}Ni_x$ alloys. Applications of The $Fe_{80}Nb_6B_{14}$ amorphous alloy as a basic precursor component for $(Fe_{80}Nb_4B_{16})_{1-x}Ni_x$ compounds can lead to forming of Fe–B matrix. This matrix can restrain magnetic long-range interaction between Fe–Ni regions, which causes decrease of magnetic moment and average hyperfine magnetic field of Fe–B–Nb–Ni investigated alloys.

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