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Influence of Cooling Rate on Magnetic Properties of $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ Type of Bulk Nanocrystalline Alloys

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The paper refers to magnetic properties of the $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ ($x = 0.06$ and $x = 0.08$) bulk nanocrystalline alloys prepared using the vacuum suction casting technique. The samples were in the form of rods with diameters of $d = 1.5$ mm, 1 mm and 0.5 mm. It was shown that for the alloys with $x = 0.08$ the significant magnetic hardening with the decrease of sample diameter was observed. For $d = 0.5$ mm the coercive field equals 2.46 T and the maximum energy product $|JH|_{max} = 77.2$ kJ/m^3.

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1. Introduction

Among well known permanent magnets, such as Co-Sm, Fe-Pt or Fe-B-Tb types of alloys and compounds [1, 2], the Fe-Nb-B-Tb alloy family plays an important role [3]. Recently we have reported that the $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ bulk nanocrystalline series of alloys can be considered as high coercive materials [4]. It was shown (Mossbauer spectra, XRD) that the examined samples contain magnetically hard $Tb_2Fe_{14}B$ and other soft $TbFe_2$, α-Fe nanocrystalline pahses. The samples with $x = 0.1$ (69% of $Tb_2Fe_{14}B$) and $x = 0.12$ (76% of $Tb_2Fe_{14}B$) are interesting. For these samples the magnetic hardening is the most significant, i.e. at $T = 300$ K, $H_c = 1.46$ T and 1.16 T, respectively. The samples were obtained by means of the so-called vacuum suction casting technique in the form of rods with diameter of $d = 1.5$ mm [5]. We concluded that the observed magnetic hardening is attributed to additional magnetic anisotropy, introduced by the casting method. Cooling rate of the preparation technique can be changed by applying moulds with different inner diameters. For the alloys with $0.1 \leq x \leq 0.12$, it was found (a work submitted for publication) that a decrease of d leads to further significant magnetic hardening, e.g. for $x = 0.12$ and $d = 0.5$ mm, $H_c = 3.36$ T and $JH_{max} = 121$ kJ/m^3. Therefore, the aim of this work is to study the influence of cooling rate on selected magnetic properties for the alloys containing less that 10 at.% of Tb.

2. Experimental procedure

The $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ ($x = 0.06$ and $x = 0.08$) bulk nanocrystalline alloys were prepared using the vacuum suction casting technique (described in [5]). The samples were in the form of rods with diameters $d = 1.5$ mm, $d = 1$ mm and $d = 0.5$ mm. Figure 1 shows a typical picture of the obtained alloys. Magnetic properties were tested by means of SQUID magnetometer (XL-7, Quantum Design) in magnetic fields up to 7 T and temperatures 10 K – 300 K.

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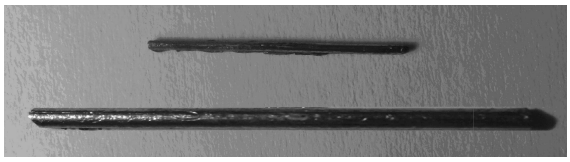

Fig. 1. Typical picture of the obtained samples ($d = 0.5$ mm, 1 mm).

3. Results and discussion

Figures 2 and 3 present magnetic hystersis loops, measured at room temperature for the alloys with $x = 0.06$ and 0.08, respectively.In the case of the alloy with 6 at.% of Tb the variety of d does not causes remarkable changes. However, for $d = 0.5$ mm a decrease of saturation magnetization was observed. In contrast to this, for the $(Fe_{80}Nb_6B_{14})_{0.92}Tb_{0.08}$ alloy the increase of the cooling rate (decrease of d) leads to significant magnetic hardening. One can observe, with decreasing d, the increase of coercive field and decrease of saturation magnetization. Let's note that for all examined values of the sample diameter, the hysteresis shapes indicate a contribution of at least two magnetic components – one relatively soft and one hard. As it is shown, the lower d, the higher

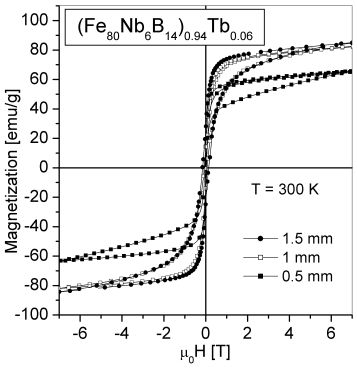

Fig. 2. Hysteresis loops for the $(Fe_{80}Nb_6B_{14})_{0.94}Tb_{0.06}$ alloy with different sample diameter.

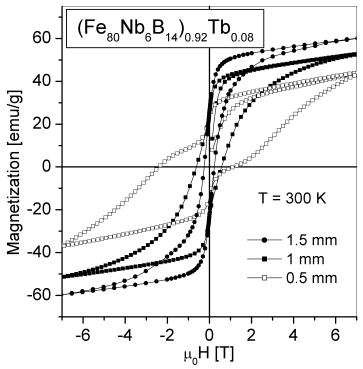

Fig. 3. Hysteresis loops for the $(Fe_{80}Nb_6B_{14})_{0.92}Tb_{0.08}$ alloy with different sample diameter.

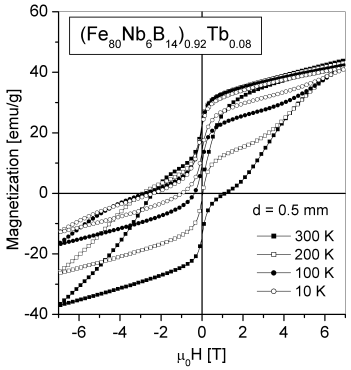

Fig. 4. Hysteresis loops for the $(Fe_{80}Nb_6B_{14})_{0.92}Tb_{0.08}$ alloy with $d = 0.5$ mm, measured at different temperatures.

contribution of the magnetic objects with hard magnetic properties is observed. The magnetic hardening is rather attributed to an additional magnetic anisotropy (shape, surface) and intergrain interactions, than to changes in magnetic phase contribution. The hysteresis (at 300 K) is asymmetric i.e. shifted in M-axis direction, which suggests the existence of some magnetic moment blocking effects, desired for permanent magnets. The blocking effects are also manifested in the temperature dependence of the hysteresis loops, obtained in one measurement sequence starting from 300 K, as shown in Fig. 4. It is clear that with decreasing temperature (lower thermal energy), more magnetic objects can be trapped in the position related to the measurement history. Therefore, the curves measured at lower temperatures are strongly shifted in M-direction. From the obtained results one can determine some characteristic quantities such as (i) the coercive fields in plus and minus H values (H_{c+}, H_{c-}), (ii) saturation magnetization (M_{s+}) and remanence (M_{r+}) in I quarter and (iii) maximum energy product ($|JH|_{max}$), that are listed in Table. Note that for the alloy with 8 at.% of Tb and $d = 0.5$ mm the coercive field reaches the value of 2.46 T and $|JH|_{max} = 77.2$ kJ/m^3.

This means that the applied vacuum suction casting allows introduction of additional magnetic anisotropy that

TABLE

Some magnetic parameters determined from the measured hysteresis loops (see the text).

Alloy x	d (mm)	H_{c-} (T)	H_{c+} (T)	M_{s+} (emu/g)	M_{r+} (emu/g)	$\|JH\|_{max}$ (kJ/m^3)
0.06	1.5	0.14	0.14	85.2	23.9	-
	1.0	0.07	0.07	82.4	14.6	-
	0.5	0.05	0.05	65.8	14.9	-
0.08	1.5	0.25	0.24	60.4	25.2	12.7
	1.0	0.62	0.57	52.5	25.4	27.4
	0.5	2.46	1.21	44.2	24.8	77.2

results in magnetic hardening. It should be underlined that the H_{c-} value increases 10 times (for $x = 0.08$), as is seen from the comparison of the alloys with $d = 1.5$ mm and 0.5 mm. The best obtained hard magnetic properties are comparable with the properties of the alloy ($d = 1.5$ mm) containing 12 at.% of Tb [4]. This means that within the alloy family and for some applications it is possible to reduce the necessary rare earth content. The physical meaning of the observed effect is nor clear at present. There are two possibilities: (i) changes in contribution of crystal phases and/or (ii) decrease of nanograins sizes, both of which are in our interest and will be studied.

4. Conclusions

Regarding the $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ bulk alloys the main conclusions can be summarized as follows.

In the case of the alloys with $x = 0.08$ the significant magnetic hardening with the decrease of sample diameter was observed. For $d = 0.5$ mm, the coercive field equals 2.46 T and maximum energy product $|JH|_{max} = 77.2$ kJ/m^3.

It was shown that the appearance of some blocking effects is responsible for the observed magnetic hardening. The origin of the effects can be in the variation of the magnetically hard $Tb_2Fe_{14}B$ nanocrystalline phase (contribution and/or size).

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