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Phase Stability of $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ Bulk Nanocrystalline Magnet

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The paper refers to phase stability of the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ bulk nanocrystalline alloys prepared using the vacuum suction casting technique. The samples were in the form of rods with diameters $d = 2$, 1.5, 1 and 0.5 mm. Heating up to 900 K reveals structural changes that occur at temperatures above 680 K (DSC and $M(T)$ measurements). The phase analysis, using Mössbauer spectra, indicates the decrease of $Tb_2Fe_{14}B$ and increase of Fe content in the samples after the heat treatment. The most stable is the alloy with $d = 1$ mm, where the formation of α-Fe phase was not observed. The decrease of d causes significant hardening i.e. coercive field increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively.

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1. Introduction

Progress in modern technologies requires new materials with specific properties for different kind of applications [1, 2]. In the field of magnetism very interesting are Fe-Nb-B type of nanocrystalline alloys [3, 4]. Recently we have reported that the $(Fe_{80}Nb_6B_{14})_{1-x}Tb_x$ bulk nanocrystalline series of alloys can be considered as highly coercive materials [5, 6]. It was shown (^{57}Fe Mossbauer spectrometry and XRD) that the examined samples contain magnetically hard $Tb_2Fe_{14}B$ and other $TbFe_2$, α-Fe soft phases, high Tb content the high contribution of $TbFe_2$. 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 significant, i.e. at $T = 300$ K, $H_c = 1.46$ T and 1.16 T, respectively. It is a characteristic feature that nanocrystalline alloys are not thermodynamically stable and, during annealing, some phase transitions (or separations) are expected to be present [7]. Therefore, in this case phase stability studies are important from application as well as scientific point of view. In this work we present structural and magnetic properties of $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ bulk nanocrystalline alloy in the as-cast state and after heating up to 900 K.

2. Experimental procedure

Samples of the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ alloy were prepared by means of the vacuum suction casting technique in the form of rods with diameters $d = 2$, 1.5, 1 and 0.5 mm (described in [8]). In the technology the variation of d causes a change of cooling. The alloys were examined before and after heating up to 900 K. Phase changes were studied using NETZSCH scanning differential calorimetry (heating rate 20 K/min), ^{57}Fe Mössbauer spectrometry (in transmission geometry with constant acceleration spectrometer, using a ^{57}Co source diffused in a rhodium matrix) and magnetic measurements (SQUID magnetometer Quantum Design XL-7 and Faraday type magnetic balance).

3. Results and discussion

Figure 1 shows an example of the DSC and thermomagnetic $M(T)$ measurements for the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ alloy with $d = 1.5$ mm. The magnetization curve clearly indicates the Curie temperature T_c (related to the $Tb_2Fe_{14}B$ phase) and structural changes (the increase of M above 680 K). The observed magnetization increase is attributed to formation of a magnetic phase with higher T_c.

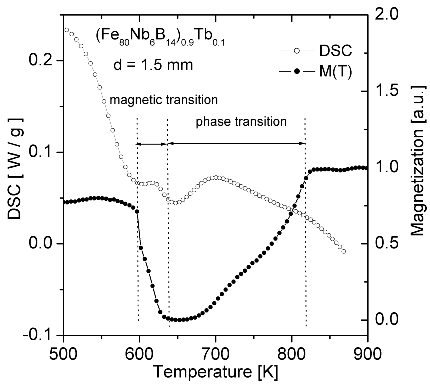

Fig. 1. DSC and $M(T)$ curves for the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ alloy with $d = 1.5$ mm.

The DSC curve reveals two exothermal peaks ascribed to the magnetic transition and phase transition (or changes). Similar results were obtained for the other

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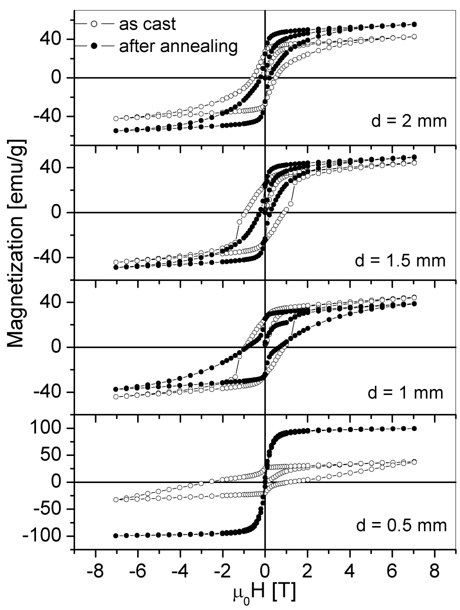

Fig. 2. Magnetic hysteresis loops measured at T = 300 K for the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ alloy with different d (before and after annealing).

examined samples with different d. Phase detection was performed based on ^{57}Fe Mössbauer spectrometry (not shown here) recorded at room temperature. For all studied cases the spectra were deconvoluted into a set of elementary Zeeman sextets (attributed to different phases) by a least-square fit procedure. The results are presented in Table.

Let's note that, except the sample with $d = 1$ mm, a decrease of $Tb_2Fe_{14}B$ and increase of α-Fe content after annealing were observed.

Figure 2 shows magnetic hysteresis loops measured at $T = 300$ K for all studied alloys before and after annealing.

TABLE

Phase content analysis (percentage), determined from Mössbauer spectra for the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ alloy. (A – as cast, B – after annealing).

d	$Tb_2Fe_{14}B$		TbFe		α–Fe		Paramagn.	
(mm)	A	B	A	B	A	B	A	B
0.5	75	17	9	14	-	53	16	16
1	65	69.5	15	15.5	-	-	14	15
1.5	62	56	15	11	6	17	17	16
2	64	56	18	11	-	17	18	16

As it is shown, the annealing causes a deterioration of hard magnetic properties (decrease of the coercive field). Apart from the main subject, for the as-cast alloys one can observe a significant magnetic hardening as a function of d (or cooling rate during sample fabrication) i.e. H_c increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively. This effect was studied in details in a separate work [9]. Generally, the change of magnetic properties is related to the phases variation. The less stable is the alloy with $d = 0.5$ mm, where the significant decrease of $Tb_2Fe_{14}B$ and separation of α-Fe was observed. In contrast to this, the most stable is the alloy with $d = 1$ mm, where in the as-cast state, as well as after heating, the formation of α-Fe was not observed. This fact has a practical meaning i.e. the annealed alloy is thermodynamically stable and possesses a good potential as permanent magnet for high temperature applications (PM motors, actuators, sensors etc.).

4. Conclusions

Referring to the $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$ bulk alloys the main conclusions can be summarized as follows.

Heating up to 900 K reveals some structural changes that occur at temperatures above 680 K (DSC and $M(T)$ measurements). The performed phase analysis, using ^{57}Fe Mössbauer spectrometry, indicates the decrease of $Tb_2Fe_{14}B$ and increase of α-Fe content for the heat treated samples. The most stable is the alloy with $d = 1$ mm, where the formation of α-Fe phase was not observed.

Regarding magnetic properties, the decrease of d causes significant hardening, i.e. the coercive field increases from 0.57 T to 2.66 T for $d = 2$ mm and $d = 0.5$ mm, respectively. The heating leads to the observed deterioration of hard magnetic properties, especially for the alloy with $d = 0.5$ mm. However for $d = 1$ mm, the alloy after annealing is quite stable and can be use in some high temperature applications.

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