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# Phase Stability of $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$ Bulk Nanocrystalline Magnet

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The paper refers to phase stability of the  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$  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  $\text{Tb}_2\text{Fe}_{14}\text{B}$  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  $\alpha$ -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  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{1-x}\text{Tb}_x$  bulk nanocrystalline series of alloys can be considered as highly coercive materials [5, 6]. It was shown ( $^{57}\text{Fe}$  Mossbauer spectrometry and XRD) that the examined samples contain magnetically hard  $\text{Tb}_2\text{Fe}_{14}\text{B}$  and other  $\text{TbFe}_2$ ,  $\alpha$ -Fe soft phases, high Tb content the high contribution of  $\text{TbFe}_2$ . The samples with  $x = 0.1$  (69% of  $\text{Tb}_2\text{Fe}_{14}\text{B}$ ) and  $x = 0.12$  (76% of  $\text{Tb}_2\text{Fe}_{14}\text{B}$ ) 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  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$  bulk nanocrystalline alloy in the as-cast state and after heating up to  $900$  K.

## 2. Experimental procedure

Samples of the  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$  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),  $^{57}\text{Fe}$  Mössbauer spectrometry (in transmission geometry with constant acceleration spectrometer, using a  $^{57}\text{Co}$  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  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$  alloy with  $d = 1.5$  mm. The magnetization curve clearly indicates the Curie temperature  $T_c$  (related to the  $\text{Tb}_2\text{Fe}_{14}\text{B}$  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  $T_c$ .

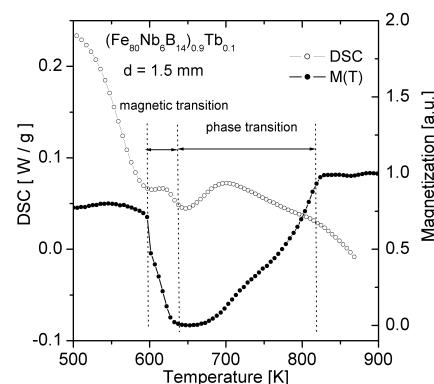


Fig. 1. DSC and  $M(T)$  curves for the  $(\text{Fe}_{80}\text{Nb}_6\text{B}_{14})_{0.9}\text{Tb}_{0.1}$  alloy with  $d = 1.5$  mm.

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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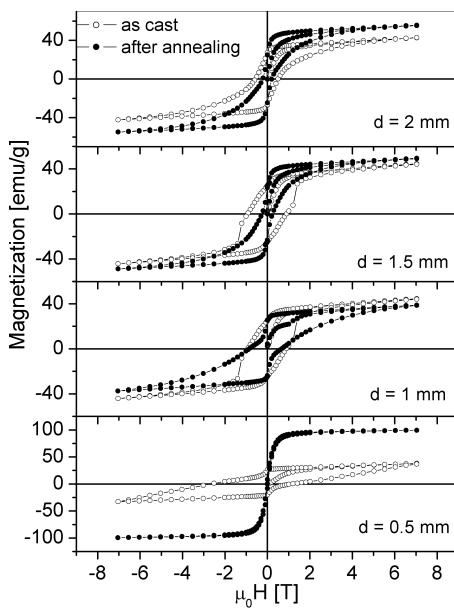


Fig. 2. Magnetic hysteresis loops measured at  $T = 300\text{ K}$  for the  $(Fe_{80}Nb_6B_{14})_{0.9}Tb_{0.1}$  alloy with different  $d$  (before and after annealing).

examined samples with different  $d$ . Phase detection was performed based on  $^{57}\text{Fe}$  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  $d = 1\text{ mm}$ , a decrease of  $\text{Tb}_2\text{Fe}_{14}\text{B}$  and increase of  $\alpha\text{-Fe}$  content after annealing were observed.

Figure 2 shows magnetic hysteresis loops measured at  $T = 300\text{ K}$  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).

$d$ (mm)	$\text{Tb}_2\text{Fe}_{14}\text{B}$		TbFe		$\alpha\text{-Fe}$		Paramagn.	
	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  $d = 2\text{ mm}$  and  $d = 0.5\text{ mm}$ , 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  $d = 0.5\text{ mm}$ , where the significant decrease of  $\text{Tb}_2\text{Fe}_{14}\text{B}$  and separation of  $\alpha\text{-Fe}$  was observed. In contrast to this, the most stable is the alloy with  $d = 1\text{ mm}$ , where in the as-cast state, as well as after heating, the formation of  $\alpha\text{-Fe}$  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  $^{57}\text{Fe}$  Mössbauer spectrometry, indicates the decrease of  $\text{Tb}_2\text{Fe}_{14}\text{B}$  and increase of  $\alpha\text{-Fe}$  content for the heat treated samples. The most stable is the alloy with  $d = 1\text{ mm}$ , where the formation of  $\alpha\text{-Fe}$  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  $d = 2\text{ mm}$  and  $d = 0.5\text{ mm}$ , respectively. The heating leads to the observed deterioration of hard magnetic properties, especially for the alloy with  $d = 0.5\text{ mm}$ . However for  $d = 1\text{ mm}$ , the alloy after annealing is quite stable and can be used in some high temperature applications.

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