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EVOLUTION OF MICROSTRUCTURE OF MELT-SPUN Nd-Fe-B ALLOY WITH LOW Nd CONTENT DURING THE HEAT TREATMENT

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ABSTRACT

In order to examine and study evolution of microstructure during the phase transformations for applied heat treatment regime for achieving the optimal values of magnetic properties of the investigated melt-spun Nd-Fe-B alloy with low Nd content were using different techniques and methods including SEM with EDS, DTA, XRD, MS analysis and magnetic measurements also.

Key words: melt-spun Nd-Fe-B, phase transformation, heat treatment, SEM, EDS, DTA, XRD, MS

1. INTRODUCTION

In recent time the study topic of many scientific institutions throughout the world is the process of obtaining high - coercive magnetic materials based on Nd-Fe-B, by the method of rapid quenching from the alloy melt of desired chemical composition, so-called "melt spinning" method.

The advantage of using the rapid quenching (R/Q) technology for obtaining high - coercive Nd-Fe-B magnets reflects in the possibility to influence through the quenching speed directly on the grain size and microstructure aimed to achieving so-called magnetic structure which provides maximal magnetic energy of finished magnetic materials of this type.

In previous research, influence of cooling rate to magnetic properties of R/Q Nd-Fe-B was investigated and optimal cooling speed for defined chemical composition was selected [1, 2].

Cooling speed range in which optimal results are achieved is rather narrow so that heat treatment of R/Q Nd-Fe-B alloys is needed in order to achieve the

maximal coercivity. For this reason the study is extended to testing of effects and selection of the most convenient regime of heat treatment for the optimal selected cooling rate and for initial defined chemical compositions of R/Q Nd-Fe-B alloy.

Magnetic material with the reduced content of Nd under the suitably selected conditions of synthesis and heat treatment has, in spite of reduced coercivity, favourable other magnetic characteristics. It is suitable for interpretation and comparison of coercitive mechanism responsible phase transformations in the function of the chemical composition and cooling rate with magnetic properties.

In this work, evolution of microstructure trough phase transformations and magnetic properties of melt-spun Nd-Fe-B alloy with Nd low content during the heat treatment was observed.

2. EXPERIMENTAL

In presented research different methods of analysis were used. The magnetic transformation and crystallization behavior were detected by differential thermal analysis (DTA).

The magnetic thermal behavior was examined using a thermomagnetic analyzer (TM). The phase identification was carried out by X-ray diffractometric analysis (XRD). Mössbauer spectroscopy (MS) was used to study the magnetic microstructure. The magnetic properties of annealed Nd-Fe-B alloy were measured using a vibrating sample magnetometer (VSM).

3.RESULTS AND DISCUSSION

Investigated Nd-Fe-B alloy with Nd low content was prepared by rapid quenching (R/Q) under Ar atmosphere [1]. SEM micrograph of melt-spun Nd-Fe-B alloy prepared at Vs = 20 m/s is shown in Fig. 1. Platelets with width between 80-140 μm and thickness between 25-35 μm are typical.

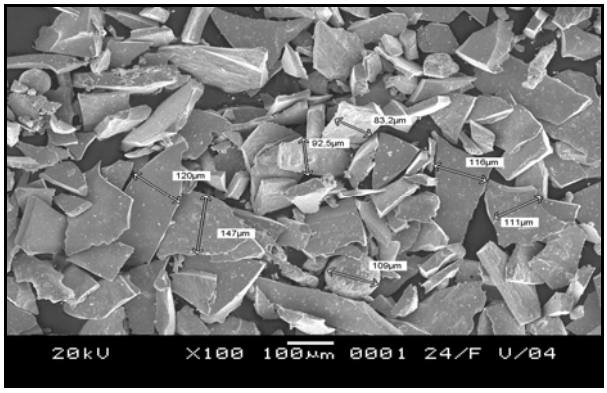


Fig.1 - SEM micrograph of Nd-Fe-B alloy

The electronic microanalysis on the energodispersion spectrometer (EDS) is used for quantitative chemical analysis. Results of chemical analysis of Nd-Fe-B sample are presented as EDS spectra shown on Fig. 2.

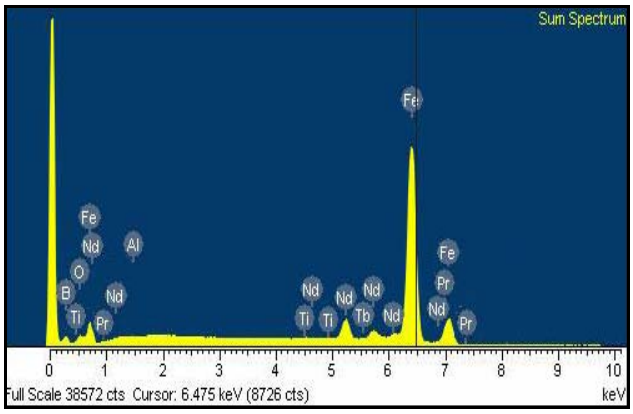


Fig. 2 - EDS spectra - chemical analysis Nd-Fe-B

Thermal behavior of investigated alloy was done by DTA in the temperature range from 20 ^{0}C to 900 ^{0}C for the reason of getting optimal heat treatment regime [3, 4].

According to the most of authors, in investigated alloy with low Nd content in temperature interval from 600^{0}C to 700^{0}C Fe_3B, $Nd_2Fe_{14}B$, $Nd_2Fe_{23}B_3$ phases and α-Fe phase are present [5, 6, 7, 8]. On the DTA curve for investigated sample (Fig.3) first crystallization peak which corresponds with primary crystallization of Fe_3B phase (phase of high magnetization) can be observed.

That is proved by XRD analysis and MS analysis [1, 4]. Second peak on DTA curve of weaker intensity corresponds with crystallization of hard magnetic phase $Nd_2Fe_{14}B$. By increasing of temperature of heat treatment in investigated interval, it was shown that density of fine Fe_3B particles is increased, and that they are uniformly distributed in the amorphous matrix for the short time of heat treatment [5, 6, 7, 8]. By analyzing of XRD diffractogram after heat treatment at 600^{0}C during 10 minutes, following phases are identified: $Nd_2Fe_{23}B_3$, Fe_3B, α-Fe and $Nd_2Fe_{14}B$ [9]. This corresponds with experimental results of DTA and with theoretical analysis of crystallization flow [7, 8, 9]. For the heat treatment at 660^{0}C during 10 minutes, same phases were identified but in different volume rations [4, 9]. Metastable phase $Nd_2Fe_{23}B_3$ crystallizes as intermediary phase, and in further process of crystallization during heating decompositions to other metastable phases: phase of high magnetization Fe_3B, main magnetic phase $Nd_2Fe_{14}B$, and small amount of soft magnetic α-Fe phase. Amount of $Nd_2Fe_{14}B$ phase comparing to Fe_3B increases with increasing of temperature of heat treatment, and that leads to increasing of coercitive force [5, 6, 7, 8]. By XRD analysis of investigated alloy heat treated at 700^{0}C during 10

minutes phases $Nd_2Fe_{14}B$, $Nd_{1.1}Fe_4B_4$, α-Fe and Fe_3B were identified. Metastable $Nd_2Fe_{23}B_3$ phase decays completely at 700^0C which proves that crystallization of identified phase has been conducted completely [7, 8, 9].

According to the results XRD and MS analysis, because of relatively high boron content, $Nd_{1.1}Fe_4B_4$ phase is also present.

On the Fig. 3 DTA curves for investigated heat treatment regime was presented.

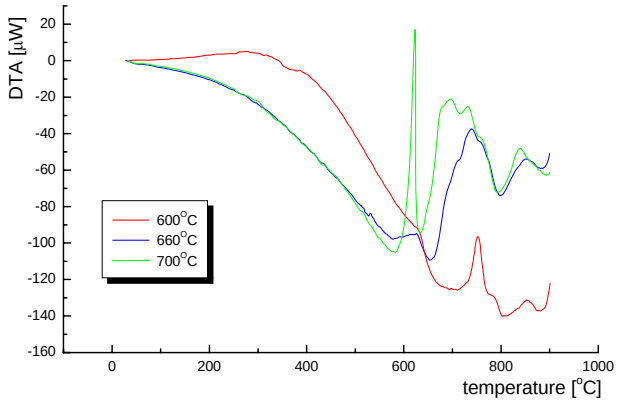


Fig. 3 - DTA curves of investigated melt-spun Nd-Fe- B alloy as a function of the annealing temperature. Annealing time: 10 min.

Corresponding Kissinger plot is shown on Fig.4.

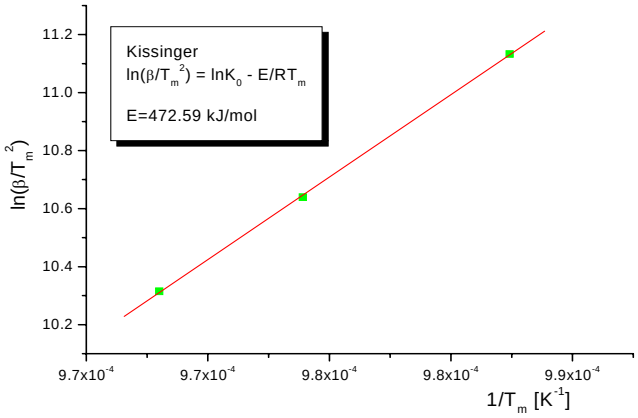


Fig.4 - Kissinger plot for Nd-Fe-B

On the basis of DTA results, Kissinger plot was used for determination of the activation energy for melt-spun Nd-Fe-B alloy with Nd low content.

Calculated value of activation energy (472.59 kJ/mol) for this Nd-Fe-B system is very close to the value that can be expected according to data found in literature [5, 6].

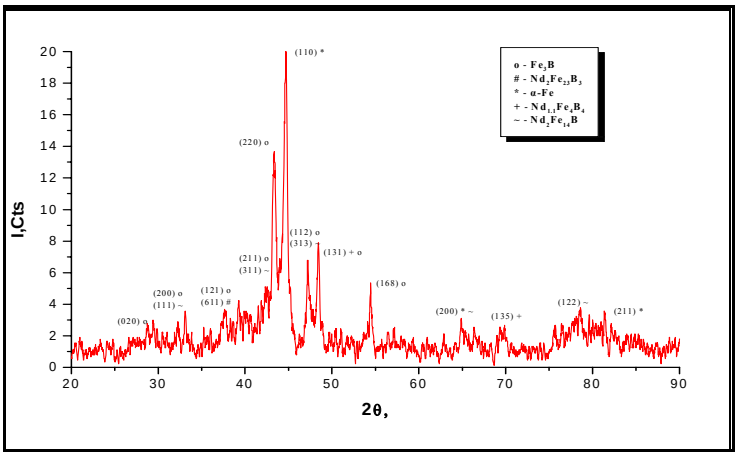


Fig. 5 - X-ray diffractogram of investigated melt-spun Nd-Fe-B annealed at 660 °C for 10 min

Mössbauer spectra were taken in the standard transmission geometry with the Co57 (Rh) source at room temperature [10]. The calibration was done against the α-iron foil data. For the spectra fitting and decomposition the CONFIT package was used [11].

For thermomagnetic measurements weakly compacted material of the cylindrical shape with a diameter of 2 mm and thickness of about 1.5 mm was used. The thermomagnetic measurement was carried out in the field of 50 Oe with the temperature sweep of 4 K/min.

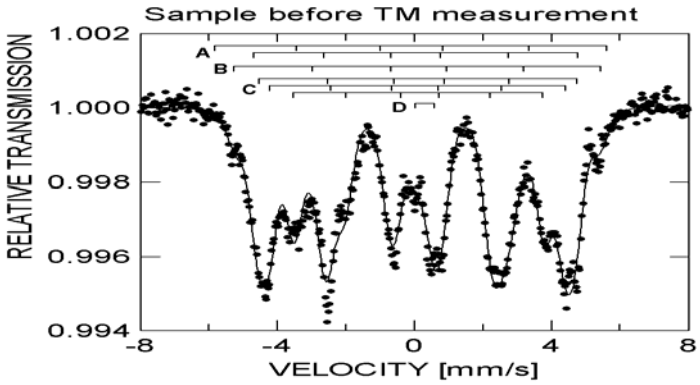
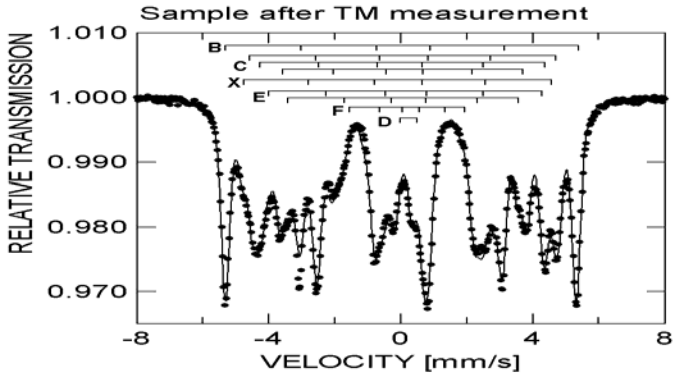


Fig. 6a



(b)

Fig. 6 - Mössbauer spectra taken before (a) and after (b) thermomagnetic measurements [A – $Nd_2Fe_{23}B_3$, B – α-Fe, C – Fe_3B, D – $Nd_{1.1}Fe_4B_4$, E – Fe_2B, F – FeB; X – belongs probably to the $Nd_2Fe_{14}B$ phase]

On the basis of previous thermomagnetic measurements in temperature range 20^0C – 800^0C after second cycle of measurements magnetic hardness of annealed specimens decreases. The Mössbauer spectra of this material after both of thermomagnetic measurement cycles are very complex and not all components could be identified exactly. MS spectra illustrate the substantial difference between the state with optimised magnetic properties (Fig.6 a) and the state after the decomposition (Fig.6 b) induced by the thermomagnetic measurement [4].

MS spectra (Fig. 6 b) exhibit the decay of the $Nd_2Fe_{23}B_3$ phase and dramatic increase content of the α-Fe phase, above all. The α-Fe and the whole set of Fe-B phases prevail. This thermal decomposition will be the main reason for the quality loss of this hard magnetic material. Magnetic measurements were performed at a room temperature on VSM with the magnetic field strength of 50 kOe.

Table 1 shows chemical composition of investigated Nd-Fe-B alloy and summary results of X-ray, MS analysis and magnetic measurements for investigated heat treatment regime.

Table 1 - Summary results of measurements

CHEMICAL COMPOSITION (MAS %)	HEAT TREATMENT REGIME	X - RAY	MOSSBAUER PHASE ANALYSIS	MAGNETIC PROPERTIES
Nd 12.0 Pr 0.2 B 4.2 Co 0.0 Al 0.3 Ti 0.0 Fe - balance	600°C	$Nd_2Fe_{14}B$ $Nd_2Fe_{23}B_3$ Fe_3B $Nd_{1.1}Fe_4B_4$ α Fe		Br 12.0 kG Hci 2.4 kOe Bh_{max} 8.0 MGOe
	660°C	$Nd_2Fe_{14}B$ $Nd_2Fe_{23}B_3$ Fe_3B $Nd_{1.1}Fe_4B_4$ α Fe	45% $Nd_2Fe_{23}B_3$ 40% Fe_3B 5% $Nd_{1.1}Fe_4B_4$ 5% unidentified phase 5% α Fe	Br 11.8 kG Hci 3.4 kOe Hcb 3.0 kOe Bh_{max} 12.0 MGOe
	700°C	$Nd_2Fe_{14}B$ Fe_3B $Nd_{1.1}Fe_4B_4$ α Fe		Br 11.0 kG Hci 3.2 kOe Bh_{max} 8.8 MGOe

Along with magnetic measurements the XRD analysis (Fig.5) and (MS) analysis (Fig. 6 a, b) were carried out to correlate microstructure and phase composition with magnetic properties for selected cooling rate and heat treatment regime [2, 3, 4]. Optimal heat treatment regime has been selected by correlation of experimental results for melt-spun Nd-Fe-B alloy.

4. CONCLUSION

Magnetic microstructure of melt-spun Nd-Fe-B alloy with Nd low content is more sensitive to thermal treatment, which has direct influence on the coercivity. Analysis of experimental results also enabled better insight in relationship between microstructure and magnetic properties in the function of heat treatment regime.

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