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MICROSTRUCTURE, MAGNETIC PROPERTIES AND THERMAL STABILITY OF MELT-SPUN Nd-Fe-B MAGNETS WITH LOW ND CONTENT

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ABSTRACT

The results of X-ray analysis, Mössbauer spectroscopy and magnetic measurement for melt-spun Nd-Fe-B with low Nd content obtained by optimal selected cooling rate and heat treatment are presented to bring some new information concerning the relation between their microstructure, magnetic properties and thermal stability.

Key words: melt spun Nd-Fe-B, microstructure evolution, magnetic properties, thermal stability

INTRODUCTION

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 cooling rate directly on the grain size and microstructure aimed at achieving magnetic microstructure which provides maximal magnetic energy of finished magnetic materials of this type [1 - 4].

Cooling rate range in which optimal results are achieved is rather narrow so that heat treatment of M/S Nd-Fe-B alloys is needed in order to achieve the maximal coercivity [2 - 4].

For this reason the study is extended to investigated effects of the most convenient regime of heat treatment for the optimal selected cooling rates for tested chemical compositions of R/Q Nd-Fe-B alloy. The evolution of microstructure in R/Q Nd-Fe-B alloys with reduced Nd content in relation to stoichiometric composition $Nd_2Fe_{14}B$ and high boron content after heat treatment was observed from the point of view of its quality as hard magnetic material with corresponding microstructure.

EXPERIMENTAL

The investigated Nd-Fe-B alloy was prepared by rapid quenching on the external surface of a copper spinning wheel under Ar atmosphere for selected optimal cooling rate corresponding to wheel speed was Vs = 15 m/s [5,6]. The

as–quenched alloy had Nd-12.0 mass %, B – 4.2 mass%, Pr-0,2 mass % and balance Fe. The heat treatment of as–quenched ribbons has been performed at 600 °C for 2 min.The X-ray diffraction(XRD) was performed using Philips diffractometar and copper anticathode (λ=0.151478nm) in the range of 2θ from 0 to 90°.

The Mössbauer spectra were taken in the transmission geometry using the Co^{57}(Rh) source at room temperature. The absorbers were prepared spreading such an amount of the material particles that the iron atoms surface density of about 10 mg/cm^2 was reached. For the spectra fitting and decomposition the CONFIT program was used [7]. The phase analysis was carried out using the data from[8 - 10]. The thermomagnetic(TM) curves completed with hysteresis loops of various initial, intermediate and final stages were measured on weakly compacted sample material of cylindrical shape with a diameter of 2 mm and thickness of about 1.5 mm. The TM measurement was done in the field 50 Oe with the temperature sweep of 4K/min. Magnetic properties in the function of the examined parameters are measured at a room temperature on the VSM with the magnetic field strength of 50 kOe.

RESULTS AND DISCUSSION

The phases present after heat treatment are given on X-ray diffractogram in Fig.1

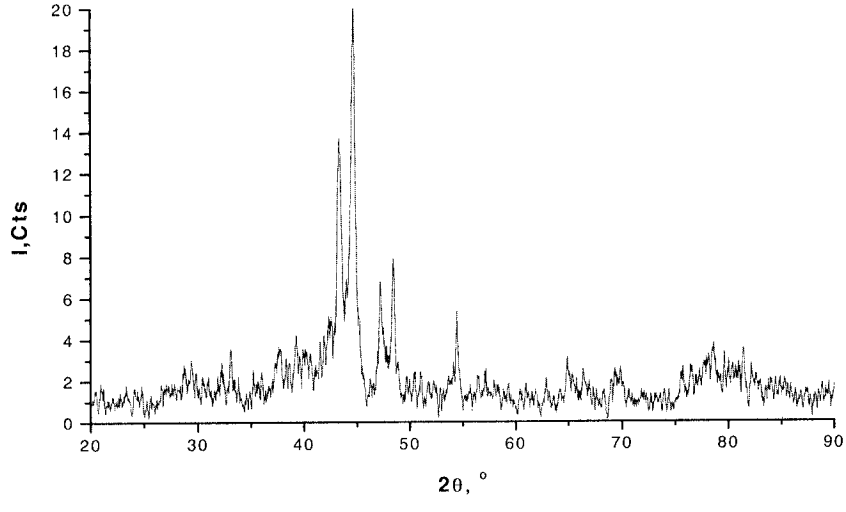

Figure 1 - The X-ray diffractogram of melt spun Nd-Fe-B, annealed at 600° C, 2 min.

According to X-ray results it has been assumed that a metastable $Nd_2Fe_{23}B_3$ crystallizes as an intermediate phase in the course of crystallization and this

phase decomposes into another metastable mixture of Fe_3B , α-Fe and $Nd_2Fe_{14}B$.With further heating the major phase is Fe_3B. The fraction of $Nd_2Fe_{14}B$ increases as composition of the alloy moves in the direction from Fe_3B to $Nd_2Fe_{14}B$ [9 - 11].

The magnetic hardness results from the structure in the form of nanometer sized particles differing in magnetic properties and many of them functioning as pinning centres for the domain wall motion. Unfortunately, such a kind of morphology with many (partially unstable) phases in nano-sized particles is very sensitive to the influence of elevated temperature [6,11]. Also here we tried to study such processes by measuring of thermomagnetic curves (Fig.2,3.)

In the Figures 4,5. the hysteresis loops before and after heat treatment of ther-momagnetic studies are presented. Already on the first run of the thermo-magnetic curve (Fig. 2), the decomposition of phases that are respondent for the hard magnetic properties of investigated material is nicely illustrated (Fig.4.). On the second run of thermomagnetic measurement (Fig.3) the antifer-romagnetic behaviour of one of the decomposition products is very interesting. This corresponds with the shape of the second hysteresis loop (Fig.5.)

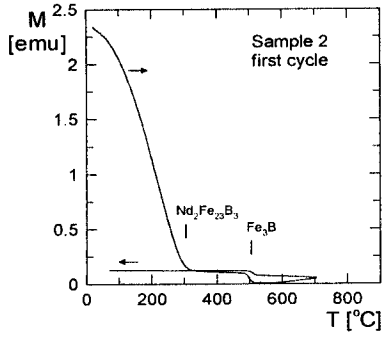

Figure 2 - First run of thermomagnetic measurement

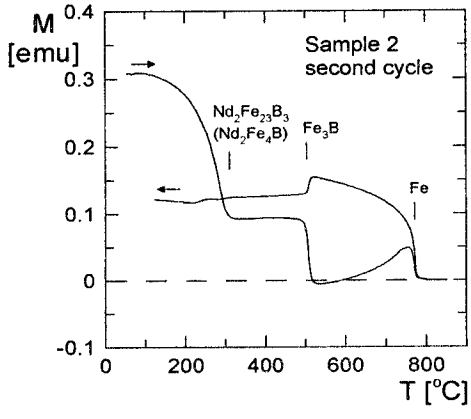

Figure 3 - Second run of thermomagnetic measurement

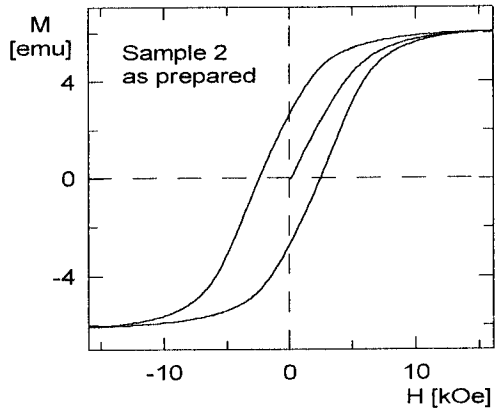

Figure 4 -Hysteresis loop before heat treatment

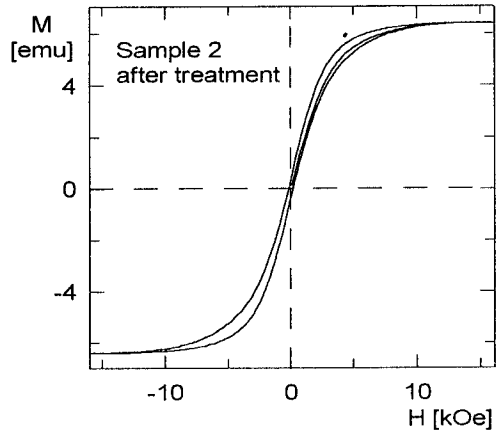

Figure 5 - Hysteresis loop after heat treatment

The Mössbauer spectra (Fig.6. a, b.) of this material after both thermo-magnetic measurement cycles are very complex and not all components could be identified exactly. MS spectra illustrate the substantial difference between the state with optimized magnetic properties and after the decomposition induced by the thermomagnetic curve measurement.

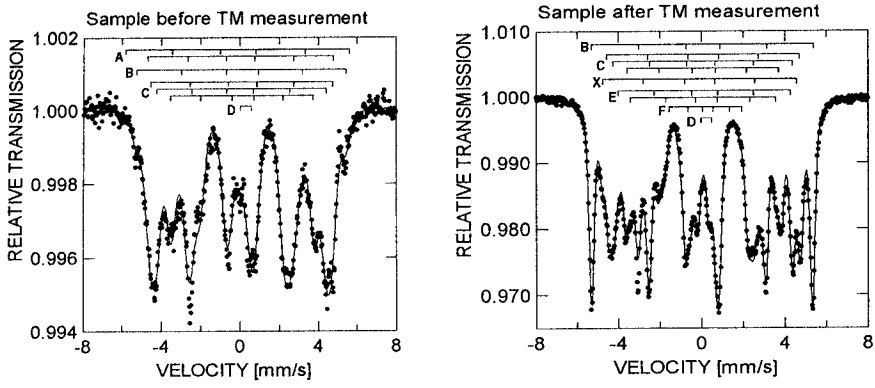

*Figure 6 - Mössbauer spectra taken before (a) and after (b) thermomagnetic measurements: (**A**- $Nd_2Fe_{23}B_3$, **B**-α-Fe,**C**-Fe_3B,**D**-$Nd_{1.1}Fe_4B_4$, **E**-Fe_2B, **F**-FeB, **X**-belongs very probably to the Nd_2Fe_{14} B phase)*

The Fig 6.b exhibits the decay of the $Nd_2Fe_{23}B_3$ phase and a dramatic increase of the content of the α-Fe phase, above all. The α-Fe and the whole set of Fe-B phases prevail. Thus the thermal decomposition will be the main reason for the quality loss of this hard magnetic material.

CONCLUSION

As an introduction to the experimental investigations of Nd-Fe-B magnet materials with low Nd content prepared by rapid quenching process for optimally selected cooling rate and heat treatment, the influence of chosen chemical composition on the evolution of microstructure, magnetic properties and thermal stability is observed.

The investigated annealed Nd-Fe-B alloy having assumed nanocrystalline composite structure with prevailing Fe_3B particles has a comparably good hard magnetic properties, but is more sensitive to thermal treatment.

The Mössbauer spectrum of this investigated alloy type after both thermomagnetic measurement cycles is very complex and not all components could be identified exactly. According to the results of XRD and Mössbauer phase analysis α-Fe and the whole set of Fe-B phases are identified. $Nd_2Fe_{23}B_3$, Fe_3B, because of relatively high boron content, $Nd_{1.1}Fe_4B_4$ are also present.

Acknowledgements

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