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# THERMOMAGNETIC BEHAVIOR AND MICROSTRUCTURE OF A RAPIDLY QUENCHED $Nd_{14}Fe_{79}B_7$ ALLOY

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## ABSTRACT
*Changes in the phase composition and crystallite size, as well as changes in the magnetic behavior of overstoichiometric $Nd_{14}Fe_{79}B_7$ alloy (32 mass% Nd), caused by the thermomagnetic measurements (TM), were observed in regard to the optimal magnetic state of this alloy.*
*In the optimized magnetic state, the hard magnetic phase $Nd_2Fe_{14}B$ is identified as the primary phase (up to 95 mass %), with a mean crystallite size about 60 nm, as determined by XRD analysis and Transmission electron microscopy (TEM). The derogation of magnetic properties after TM is due to decreased amount of $Nd_2Fe_{14}B$ phase, the formation of $Nd_2O_3$ and different Fe(O)B phases, as well as an increase in the mean crystallite size (e.g. $Nd_2Fe_{14}B \approx 95$ nm).*
**Keywords:** Nd rich Nd-Fe-B alloy, TM-measurement, phase composition, crystallite size, magnetic properties

## 1. INTRODUCTION
The rapid quenched Nd-Fe-B alloys with overstoichiometric Nd content have an almost monophase microstructure with dominant amount of $Nd_2Fe_{14}B$ hard magnetic phase and small amount of Nd rich phases situated at the $Nd_2Fe_{14}B$ grain boundaries. [1]
The magnetic properties of these alloys are strongly dependent of phase composition, grain size of presented phases and their distribution [1-3]. This paper covers part of the experimental results of changes in the phase composition and crystallite size and consequently changes in the magnetic behaviour of investigated $Nd_{14}Fe_{79}B_7$ alloy induced by thermomagnetic measurements (TM) up to 800°C. The mentioned changes were observed in regard to the optimal magnetic properties of the alloy after optimal heat treatment (630°C, 3 min).

## 2. EXPERIMENTAL
The $Nd_{14}Fe_{79}B_7$ alloy was produced by centrifugal atomization and its magnetic characteristics in the optimized magnetic state were ${}_iH_c$ = 16.2 kOe (1296 kA/m), $B_r$ = 7.4 kG (0.74 T) and $(BH)_{max}$ = 10.6 MGOe (85 J/m$^3$). TM measurements in the temperature interval 20–800°C were performed using a vibrating sample magnetometer in a field of intensity of 50 Oe under vacuum. The phase composition and crystallite size in the optimized magnetic state and after TM were determined by XRD analysis using an X'Pert PRO MPD diffractometer from PANanalytical with Co $K_\alpha$ radiation. The size-strain

analysis and quantitative phase analysis of the obtained XRD data were realised using FullProf software [4]. The X-ray line broadenings were analyzed through refinement of the TCH-pV function parameters. The microstructure of the alloy in the optimized state was observed by TEM. Hysteresis loops before and after TM was obtained using SQUID with a magnetic field strength of 50 kOe.

**3. RESULTS AND DISCUSSION**

The thermomagnetic behaviour of $Nd_{14}Fe_{79}B_7$ alloy and the corresponding phase transformations were observed by TM measurements. The obtained TM curves are presented on Fig. 1.

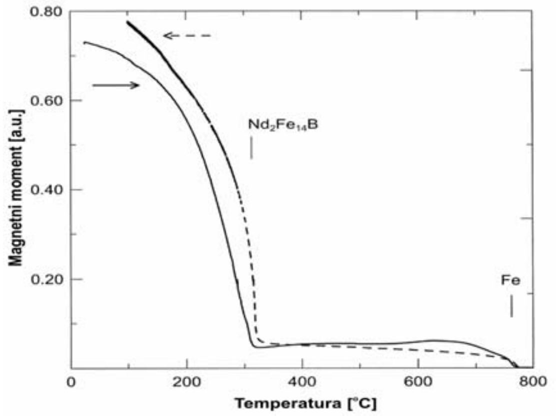


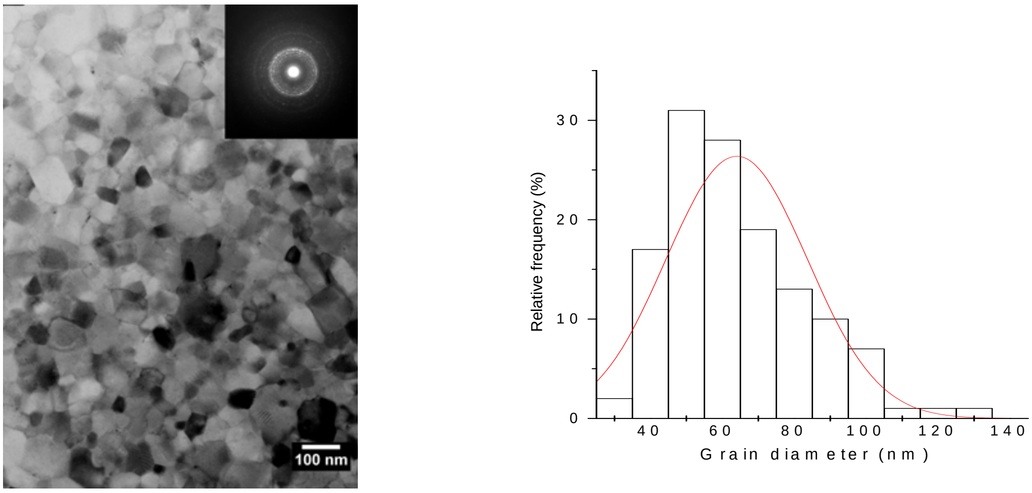

*Figure 1. TM curves of the investigation $Nd_{14}Fe_{79}B_7$ alloy*

For better understanding of the influence of content and grain size of individual phases on the magnetic properties before and after the TM, the size-strain and quantitative phase analyses of XRD data were done. Summarized results are presented in Table 1.

*Table 1. Phase composition with mean crystallite size for the $Nd_{14}Fe_{79}B_7$ alloy before and after thermomagnetic measurement obtained by size strain analysis*

| Phases | Before thermomagnetic measurement | | After thermomagnetic measurement | |
|---|---|---|---|---|
| | **Amount** [%] | **Crystallite size** [nm] | **Amount** [%] | **Crystallite size** [nm] |
| $Nd_2Fe_{14}B$ | 95 | 57 | 75.0 | ~ 63.0 |
| α - Fe | 5 | 59 | 3.35 | ~ 64.0 |
| $Nd_2O_3$ | - | - | 5.7 | ~ 17.1 |
| $Fe_{17}Nd_5$ | - | - | 13.8 | ~ 16.4 |

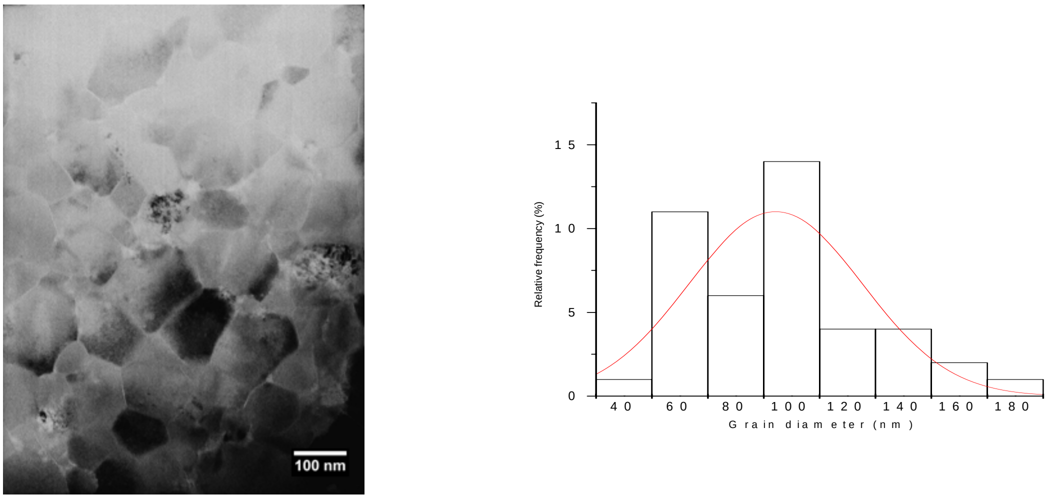
In the optimized magnetic state the hard magnetic phase $Nd_2Fe_{14}B$ is identified as the primary phase (up to 95 mass %) with a mean crystallite size about 57 nm and presence of α-Fe phase (5 mass %) with mean crystallite size of 59 nm. The TEM micrograph (Fig. 2,), showing the average grain size in the optimized magnetic state about 64 nm, confirms the mean crystallite size determined by XRD analysis. (≈60 nm)




Average = 64 nm; St.Dev. = 19.66

*Figure 2. TEM micrograph of $Nd_{14}Fe_{79}B_7$ alloy before TM measurement (in optimal magnetic state) with grain size distribution*

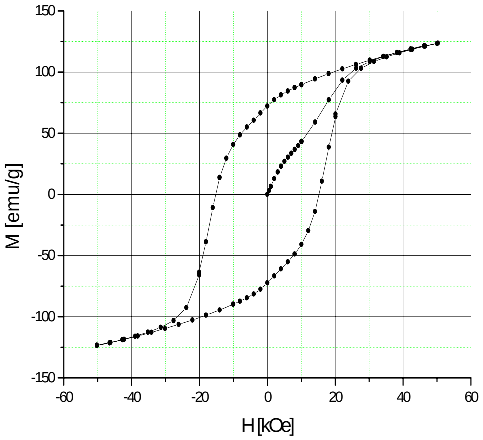
Regarding the $Nd_2Fe_{14}B$ phase, the decrease of the amount and the increase of grain size of hard magnetic phase after the TM measurement are obvious. Its content is estimated to 75 mass%, with mean grain size about 64 nm. The presented TEM micrograph (Fig 3.) shows the increase of grain size of present phases after TM measurements, as well.




Average = 94 nm; St.Dev = 31.154

*Figure 3. Transmission electron micrograph of $Nd_{14}Fe_{79}B_7$ alloy after thermomagnetic measurement with grain size distribution*

Greater grain growth observed by the TEM analysis compared to that determined by the XRD analysis can be explained by the different crystallinity of investigated phases and different nature of the applied methods. Measurements of magnetic properties in the state before (in optimal magnetic state) and after TM, carried out on the SQUID magnetometer are illustrated by SQUID hysteresis loops presented on Fig 4.



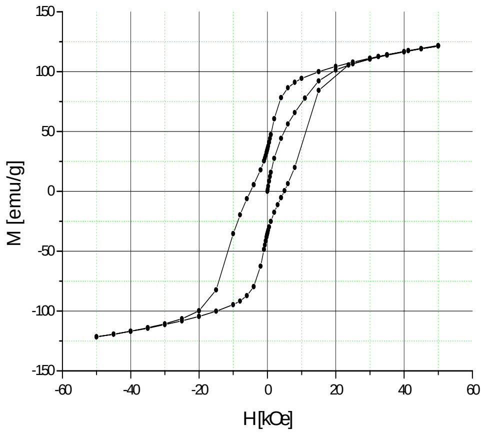




*Figure 4. SQUID hysteresis loops of $Nd_{14}Fe_{79}B_7$ alloy before and after TM measurement*

The phase transformations which have occurred during TM measurement and the growth of crystal grains are in correspondence with a quality loss of the hard magnetic properties after TM.

## 4. CONCLUSION

In the light of presented facts, it can be concluded that the structural and microstructural changes (phase compositions, crystallite size) of the investigated Nd-Fe-B alloy have a direct influence on the magnetic behavior and they are the main reason for the deterioration of magnetic properties after TM measurements.

## 5. REFERENCES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