COERCIVITY AND MECHANICAL PROPERTIES OF Nd-Fe-B MAGNETS IN DEPENDENCE ON THE AVERAGE GRAIN SIZE

W. RODEWALD, M. KATTER AND K. ÜSTÜNEN
Vacuumschmelze GmbH & Co. KG, P. O. B. 2253, 63412 Hanau, Germany
Corresponding author e-mail: werner.rodewald@vacuumschmelze.com

Abstract
The coercivity and some mechanical properties of sintered (Nd,Dy)-Fe-TM-B magnets, TM: Co, Cu, Al, Ga, have been examined at room temperature in dependence on the fraction of RE-rich constituents and on the average grain size. The increase of the coercivity $H_c$ of fine grained magnets, represented by a potential regression curve $y = \alpha x^\beta$, may be due to reduced local demagnetizing fields and a smaller probability of the nucleation of reversed domains. The compressive and bending strengths amount to $(960 \pm 50)$ N/mm² or $(330 \pm 20)$ N/mm², respectively, and do not depend on the grain size significantly. The fracture toughness ranges between 180 and 200 Nmm$^{-3/2}$. From ultrasound attenuation a Young’s modulus of about $160\text{kN/mm}^2$ was deduced. The Vickers hardness amounts to $(610 \pm 30)$ HV for magnets with average grain sizes between 4 and 9.5 µm.

Introduction
Sintered Nd-Fe-B magnets are applied in many devices, for instance motors, generators, couplings, bearings, separators, sensors, etc. in order to optimize the efficiency or in order to reduce the weight or volume of the magnet assemblies. In general magnets operate as functional materials, but in dynamic applications also mechanical properties may become important.

Some mechanical properties of sintered Nd-Fe-B magnets have been examined in dependence of different processing routes [1], of the composition, for instance the Co-content [2, 3] or different RE-rich constituents [4] or various additions of Al, Cr, Nb, Zr [3, 5].

Besides the composition the mechanical properties may depend on the microstructure, characterized by the average grain size. This report presents results on the magnetic as well as on the mechanical properties of sintered Nd-Fe-B magnets in dependence on the average grain size.

1 Preparation of the test samples
Sintered (Nd,Dy)-Fe-TM-B magnets, TM: Co, Cu, Al, Ga, were prepared from ingot materials by powder metallurgy. The microstructure of anisotropic Nd-Fe-B magnets consists of hardmagnetic Nd$_2$Fe$_{14}$B grains, non-magnetic Nd$_4$Fe$_4$B grains, Nd-rich constituents and impurities, such as pores, Nd-oxides etc. In order to investigate the mechanical properties in dependence on the fraction of RE-rich constituents, sintered Nd$_{12.8-x}$Dy$_{0.74}$Fe$_{79.52-x}$TM$_{1.36}$B$_{5.58}$ magnets, TM: Co, Cu, Al, Ga, $x = 0, 0.1, 0.3, 0.9, 1.4, 2.0$, have been prepared. Due to the different amounts of liquid phase at a sintering temperature of $1100^\circ\text{C}$, the average grain size of the magnets varies between 6.5 and 9.5 µm.

In an additional test Nd$_{12.9}$Dy$_{0.74}$Fe$_{79.52}$TM$_{1.26}$B$_{5.58}$ magnets were sintered from alloy powders with different average particle sizes, for instance 2.1, 2.6, 3.1, 3.5, 3.7 and 4.1 µm according to FSSS. By adjusted sintering conditions, dense magnets with an average grain size between 4.0 and 6.5 µm could be achieved.

The density of the sintered magnets was controlled by the Archimedes principle. The average grain size has been determined by the three circular intercept method, according to the standard ASTM E112. The magnetic properties are deduced from demagnetization curves, measured by a hysteresisgraph at different temperatures. In particular the dependence of the coercivity $H_{ci}$ and of the temperature coefficient $TC(H_{ci})$ on the average grain size has been examined.

The following mechanical properties were determined by standardized tests: the compressive and the bending strength, the fracture toughness, the Young’s modulus, and the Vickers hardness.
2 Dependence of the magnetic and mechanical properties on the average grain size of sintered (Nd,Dy)-Fe-TM-B magnets

Due to liquid phase sintering, (Nd,Dy)-Fe-TM-B magnets achieve easily densities between 7.55 and 7.6 g/cm³ or $\rho/\rho_0 > 99\%$, respectively. The typical magnetic properties amount to $B_r = (1.42 \pm 0.02)\ T$ and $H_{cj} = (12 \pm 1)\ kA/cm$. The maximum energy densities range between 360 and 400 kJ/m³.

2.1 Coercivity $H_{cj}$ and temperature coefficients $TC(H_{cj})$

The coercivity of sintered (Nd,Dy)Fe-TM-B magnets is determined by the magnetocrystalline field strength, reduced by local demagnetizing fields \([6 - 8]\). The local fields depend on the shape and the volume of the reversed grains. Hence in magnets with a reduced grain size, the local fields and the probability of the nucleation of reversed domains will be smaller, what results in increased coercivities $H_{cj}$, see Fig. 1. The potential regression curve demonstrates, that the coercivity of $\text{Nd}_{12.9}\text{Dy}_{0.74}\text{Fe}_{79.52}\text{TM}_{1.26}\text{B}_{5.58}$ magnets increases by

$$H_{cj}(20\,^{\circ}C) = \text{const} \cdot (\text{grain size } D \text{ in } \mu m)^{-0.19} \quad 4\,\mu m < D < 10\,\mu m \quad (1)$$

with decreasing grain size.

![Figure 1: Coercivity $H_{cj}$ of $\text{Nd}_{12.8-x}\text{Dy}_{0.74}\text{Fe}_{79.52-x}\text{TM}_{1.36}\text{B}_{5.58}$ magnets, TM: Co, Cu, Al, Ga, $x = 0, 0.1, 0.3, 0.9, 1.4, 2.0$ and of $\text{Nd}_{12.9}\text{Dy}_{0.74}\text{Fe}_{79.52}\text{TM}_{1.26}\text{B}_{5.58}$ magnets in dependence on the average grain size. The potential regression curve demonstrates an increase of the coercivity $H_{cj}$ with decreasing grain size.](image)

According to the experimental results the temperature coefficient $TC(H_{cj})$ for temperatures between 20 and 100 °C is reduced for magnets with a decreased average grain size, see Fig. 2. Hence sintered Nd-Fe-TM-B magnets with a fine-grained microstructure may have an improved temperature stability, if the squareness of the demagnetization curve $J(H)$ has remained constant.
2.2 Compressive strength

The compressive strength has been measured on cubes with dimensions of approximately 5 mm x 5 mm x 5 mm, which were cut from sintered (Nd,Dy)-Fe-TM-B magnets parallel to the easy axis by diamond grinding. On the average the compressive strength amounts to (960 ± 50) N/mm² and does

![Figure 3: Compressive strength of Nd₁₂.₈₋ₓDyₓFe₇₉.₆₋ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂xlim=x_ninex/10,x/10,etc. magnets, TM: Co, Cu, Al, Ga, x = 0, 0.1, 0.3, 0.9, 1.4, 2.0 and of Nd₁₂.₉Dy₀.₇₄Fe₇₉.₅₂₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂ₓₐₐ₄₅₂xlim=x_ninex/10,x/10,etc. magnets in dependence on the average grain size. The dashed graphs represent the compressive strength $f_{\text{comp}} \pm \sigma$, $\sigma$ denotes the standard deviation.
not depend on the grain size of the sintered magnets within the accuracy of the measurements, see Fig. 3. These results are in fair agreement with measurements on Nd-Dy-Fe-Co-B magnets by Rabinovich [3]. Measurements of the compressive strength perpendicular to the easy axis result in about 4 to 8 % smaller values.

### 2.3 Bending strength

The bending strength has been measured on bars with dimensions of approximately 5 mm x 2 mm x 50 mm, according to the standard ASTM 314-64. On the average the bending strength perpendicular to the easy axis amounts to about \((330 \pm 20) \text{ N/mm}^2\) and does not depend on the grain size of the sintered magnets within the accuracy of the measurements, see Fig. 4. There is a small anisotropy of the bending strength. In general the bending strength perpendicular to the easy axis results in 4 to 10 % smaller values than parallel to the easy axis. Within the accuracy of these measurements on sintered Nd-Fe-Co-B materials, the results are in fair agreement with data of J. Jiang et al. [2] and Yu. Rabinovich et al. [3].

In addition the bending strength has been measured on magnetized bars perpendicular to the easy axis, what results in about 10 to 15 % smaller values. The repulsive magnetic forces promote the crack formation and propagation.

![Figure 4: Bending strength of NdFeB magnets in dependence on the average grain size.](image)

**Figure 4:** Bending strength of Nd$_{12.8-x}$Dy$_{0.74}$Fe$_{79.52-x}$TM$_{1.36}$B$_{5.58}$ magnets, TM: Co, Cu, Al, Ga, x = 0, 0.1, 0.3, 0.9, 1.4, 2.0 and of Nd$_{12.9}$Dy$_{0.74}$Fe$_{79.52}$TM$_{1.26}$B$_{5.58}$ magnets in dependence on the average grain size. The dashed graphs represent the bending strength, \(\sigma\), \(\sigma\) denotes the standard deviation.

### 2.4 Fracture toughness

The fracture toughness has been examined on bars, 3 mm x 6 mm x 30 mm, which have got a notch of 0.2 mm in width and 3 mm in length in the middle of the bar, made by spark erosion wire cutting. The fracture force was determined by a three point bending test according to ASTM 314-64. The cracks always started at the notch and extended symmetrically. On the average the fracture toughness perpendicular to the easy axis amounts to \((184 \pm 10) \text{ N/mm}^{3/2}\). There is only a small decrease of the fracture toughness with decreasing grain size, see Fig. 5. A small increase of the Nd-rich constituents does not affect the fracture toughness significantly.

Within the accuracy of the measurements the results confirm the data on the fracture toughness of sintered Nd-Dy-Fe-B magnets by the Chevron notch bend test, J. A. Horton et al. [1, 9].
$K_{IC} = 7,036 \text{ (grain size } D \text{ in } \mu\text{m}) + 147$

Figure 5: Fracture toughness $K_{IC}$ of Nd$_{12.9}$Dy$_{0.74}$Fe$_{79.52}$TM$_{1.26}$B$_{5.58}$ magnets, TM: Co, Cu, Al, Ga, sintered of alloy-powders with different average particle sizes at adjusted temperatures, in dependence on the average grain size. The dashed graphs represent the fracture toughness $K_{IC} \pm \sigma$, $\sigma$ denotes the standard deviation.

2.5 Young’s modulus

The Young’s modulus has been determined on bars, 3 mm x 4 mm x 50 mm, by ultrasound attenuation. For sintered Nd$_{12.9}$Dy$_{0.74}$Fe$_{79.52}$TM$_{1.26}$B$_{5.58}$ magnets with an average grains size of 9.8 µm the Young’s modulus amounts to (160 ± 3) kN/mm$^2$ perpendicular to the easy axis. The results confirm the data on sintered Nd-Dy-Fe-Co-B magnets by Yu. M. Rabinovich et al. [3].

Measurements of the Young’s modulus parallel to the easy axis result in similar values. Within the accuracy of the measurements, there is only a negligible anisotropy (<3 %) of the Young’s modulus.

2.6 Vickers hardness

The Vickers hardness has been examined on polished surfaces of Nd$_{12.8-x}$Dy$_{0.74}$Fe$_{79.52-x}$TM$_{1.36}$B$_{5.58}$ magnets, TM: Co, Cu, Al, Ga, $x = 0, 0.1, 0.3, 0.9, 1.4, 2.0$, and on Nd$_{12.9}$Dy$_{0.74}$Fe$_{79.52}$TM$_{1.26}$B$_{5.58}$ magnets by a load of 10 N. There is no significant dependence of the Vickers hardness on the average grain size, see Fig. 6. This may be due to the wide distribution of the grain sizes in sintered magnets. For instance even magnets with an average grain size of about 5 µm still contain some large grains with dimensions up to 20 µm. On the average the Vickers hardness amounts to (610 ± 30) HV, what is about 10 % higher than the Vickers hardness measured by J. Jiang et al. [2] and Yu. Rabinovich et al. [3].
3 Conclusions

The coercivity $H_{cJ}$ of sintered (Nd,Dy)-Fe-TM-B magnets, TM: Co, Cu, Al, Ga, increases for magnets with a smaller grain size, what agrees with the investigations by Y. Kaneko [10]. Due to the smaller grain size the demagnetizing fields of the reversed grains are decreased and the probability for nucleation of reversed domains is reduced. As a consequence the coercivity of the sintered magnets is enhanced. Simultaneously the temperature coefficient $TC(H_{cJ})$ decreases, what promotes the temperature stability of fine grained magnets.

The compressive or the bending strengths of sintered (Nd,Dy)-Fe-TM-B magnets amount to $(960 \pm 50)$ N/mm² or $(330 \pm 20)$ N/mm² and do neither depend on the average grain size nor on the fraction of RE-rich constituents within the accuracy of these measurements. There is no significant anisotropy of the compressive or of the bending strength.

The fracture toughness of sintered (Nd,Dy)-Fe-TM-B magnets ranges between 180 and 200 Nmm$^{-3/2}$. Since the fracture occurs mainly intergranular, the average grain size does not affect the fracture toughness significantly.

The Young’s modulus amounts to $(160 \pm 3)$ kN/mm² for sintered (Nd,Dy)-Fe-TM-B magnets with an average grain size of 9.8 µm. The anisotropy of the Young’s modulus parallel or perpendicular to the easy axis is less than 3 %.

The Vickers hardness ranges between 580 and 640 HV. Due to the wide grain size distribution in sintered magnets, there is no significant dependence of the Vickers hardness on the average grain size of sintered (Nd,Dy)-Fe-TM-B magnets.

Acknowledgements

The authors are grateful to S. Stein and J. P. Jacquet for the technical assistance in preparing sintered magnets and in performing the tests.
References