# THE CHARACTERIZATION OF GRINDED NdFeB MAGNETIC MATERIALS OBTAINED FROM ELECTRIC MOTORS

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**Abstract**

The hard magnetic materials play an important role in modern technology, e.g. automotive, electronics and many daily use equipment. The overview of statistics indicates that over the next few years the continuous increase of their consumption will be observed. Currently, China is the largest importer of rare earth elements, from which RE-M (rare earth elements, transition metal) magnets are produced. This situation is inconvenient for European countries because of their dependence on the largest producer. That aspect combined with high material and production costs makes the investigation into possibilities of effective recycling methods of hard magnets a research area of significant importance.

In the paper the characteristic of magnetic composite material based of RE-M-B (rare earth elements, transition metal, boron) powder has been presented. For the research, the new magnetic material had been prepared from recycled hard magnetic elements (from electric motors) and the plastic as a binding material. The magnetic material characteristic includes, a brief outline of the recovery technology (treatment and processing of recycled permanent magnets) as well as a package of materials research analysis. The paper brings the opportunities to introduce the hard magnets recycling technology on an industrial scale.

**Keywords:** NdFeB magnets, recycling, rare earth elements

## 1. INTRODUCTION

Currently, the magnetic materials occupy an extremely important position in many areas of life, are decisive for the existence of modern civilization and its development. Commonly, all known elements and chemical compounds based on magnetic properties are classified as magnetic materials. In practice, the most applicable are ferrous metals. Among them magnetic hard materials are out standing to be used as permanent magnets. Nowadays, the magnetic materials market focuses on rare earth (RE) and transitional (M) metals-based materials, thus the allow the creation of permanent magnets with high magnetic properties. Due to a high level of magnetic energy of these magnets they give the ability for significant miniaturization of electronic and mechanical devices. A variety of technical products contain neodymium magnets, such as speakers, hard drives, electric motors and hybrid cars [1]. In connection with the last development of electronics and the application of neodymium magnets in electromagnetic motors this kind of material becomes more and more common. The use of hybrid engines in cars is the basis for the reduction of harmful gas emissions while, on the other hand it contributes to a growth of problematic waste such as the used neodymium magnets. Another challenge that is faced by manufacturers of electric motors are resources of these elements that are running out and their largest and nearly sole exporter China [2, 3]. Mining of the rare earths is complex and in the process radioactive waste is produced. Currently, only a small part of the neodymium magnets is recovered and effective methods of processing of these materials are still being sought. Production of great quantities of waste by modern population requires determined action in the form of recycling and reuse of raw materials - in particular, this is applied to materials containing rare elements [4 - 9].

## 2. EXPERIMENTAL

Due to the constant increase of electro-waste and growing demand for magnetic materials, in this work the preparation of the proposed recycling technology has been presented. This paper consists of the general characteristics of magnetic materials (**Figure 1**), in this case neodymium magnets obtained as a result of the dismantling of hybrid engines. The preparatory action is the release of the magnets from a until, in which they are fitted with glue residue binder (resin). Demagnetization is possible through a series of thermal processes. In the case of the tested neodymium magnets temporary demagnetization has been taken place in temperatures approaching 350 °C, which is the so-called Curie temperature (magnetic material looses its unique magnetic properties).


**Figure 1** Neodymium magnets recovered from electric motors and powder after melting in three different times: a) 10 s, b) 15 s, c) 30 s

Neodymium magnets (RE-M-B-type) are characterized by low corrosion resistance to external environment which limits their application and shortens the life of equipment [5]. The low corrosion resistance is related to the contents of the chemical elements belonging to rare earth group (up to 30% of the mass in the case of neodymium magnets). Magnetic properties of Nd-Fe-B magnets depend on their chemical composition and processing technology [10-14].

### 2.1. Material preparation-demagnetization and fragmentation of magnets

The study has been performed on samples of neodymium magnets in the recycling process of used motors. Taking into account the multi-phase structure of the magnets and neodymium high affinity to oxygen, the preparation of materials has been conducted in a noble gas atmosphere (Ar). For demagnetization, the magnetic material has been heated in an oven to a temperature above the Curie temperature. Subsequently grinding in ball mill has been performed, keeping in mind that excessive fragmentation of the powder will reduce the magnetic coercivity, which in turn would result in the loss of magnetic properties of the starting material [11,12].

The variable parameter during grinding of the powder was time. The three grinding times have been applied: 10, 15 and 30 seconds. The milling process has been done at the automatic milling machine. An important phenomenon affecting the properties of the material being ground is the alteration of crystallographic structure in the case of neodymium magnets. Prolonged grinding leads to deformation of crystalline and to recombination of the phase - the crystallographic structure of the ferromagnetic phase is damaged [11-14].

### 2.2. Research methodology

In the course of the analysis special caution was maintained during preparation of magnetic materials in order to prevent autoignition (spontaneous combustion), which occurs by means of sudden reactions of rare earth elements with oxygen [11]. These processes were carried out under the cover of a noble gas of argon. Demagnetization temperature did not exceed 360°C, which protected the magnets from a permanent demagnetization [13]. In the experimental part a microscopic analysis, the analyses of the chemical composition of neodymium magnets from hybrid engines as well as a particle size analysis have been presented.

## 3. RESULTS AND DISSCUSION

### 3.1. Microscopic analysis

The study was performed on demagnetization samples. The grain boundary and structure were revealed by etching the surface with Nital. The composition of the etching substance included: Nitric acid 5 $cm^3$ and 100 $cm^3$ Ethanol. During the tests of microscopic analysis of the magnetic material three phases were observed. As the typical NdFeB-magnets structure consists of three phases: the ferromagnetic phase $Nd_2Fe_{14}B$ ($\Phi$) about 85% of the phase volume, the boron-rich phase $NdFe_4B_4$ ($\eta$) about 3% and the neodymium-rich phase NdFe about 12% mass [16-18]. Neodymium magnets are structured in the multi-phase skeletal manner, i.e. phases rich in neodymium fill intergranular areas around grains of $Nd_2Fe_{14}B$ phase. Individual phases can be observed in **Figure 2**.

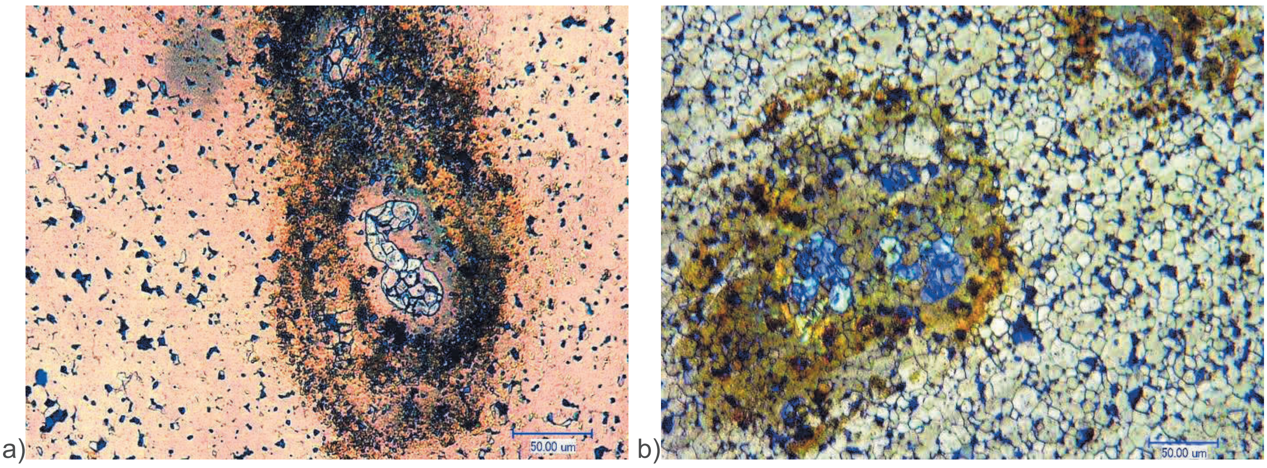


**Figure 2** The microstructure images of RE-M-B magnetic material recovered from electric motors

### 3.2. Chemical composition of the magnetic powder for laboratory tests

By analyzing the chemical composition the detailed analysis of the content of individual elements has been obtained. For chemical analysis was used atomic emission spectroscopy ICP-OES Vista MPX, Varian. **Table 1** shows the results of the analysis of the chemical composition of the RE-M-B alloy. They indicate that the tested neodymium magnets, apart from the base alloy component, neodymium (ca. 24%), the presence of other rare earth elements was identified Dy (ca. 6%), and Pr as well as Tb. Whereas the Fe substituents in the tested alloys are also Co, Al, Pb, Cu and others. Thus in the tested material was identified RE = Nd, Dy, Pr, Tb and M = Fe, Co, Al, Pb, Cu.

**Table 1** The chemical composition of the RE-M-B magnetic material recovered from electric motors - determined with atomic emission spectroscopy

| *Chemical composition* | *Content [%]* | *Chemical composition* | *Content [ppm]* |
|---|---|---|---|
| B | 0.88 | Al | 3301 |
| Co | 1.03 | Ca | 129 |
| Dy | 5.37 | Cr | 116 |
| $Fe_{ges}$ | 62.06 | Cu | 985 |
| $Fe_{met}$ | 59.29 | Mn | 291 |
| Nd | 23.56 | Pb | 1729 |
| Pr | 0.81 | | |
| Tb | 0.12 | | |

### 3.3. Particle size analysis

The powder characteristics have been studied in order to determine the size. The morphology of the powders obtained after the shredding process has a significant impact on the technological qualities of powders. The analysis has been performed with the use of laser diffractometer HELOS wet, Sympatec in the particle range 0.1 microns to 8750 microns. The measuring element was placed in the Fourier plane (focal length lenses) set of sensors (detectors), to measure the light intensity in the polar coordinates of the high resolution. An analysis of transforming images detects repetitive grains, even when regularity is not noticeable to the naked eye. In this analysis, the same grains give the characteristic stripes or points, and their intensity is proportional to the content. In the process of grinding three groups of powders granulation have been obtained (depending on the grinding duration time) - shown in **Figure 3**.

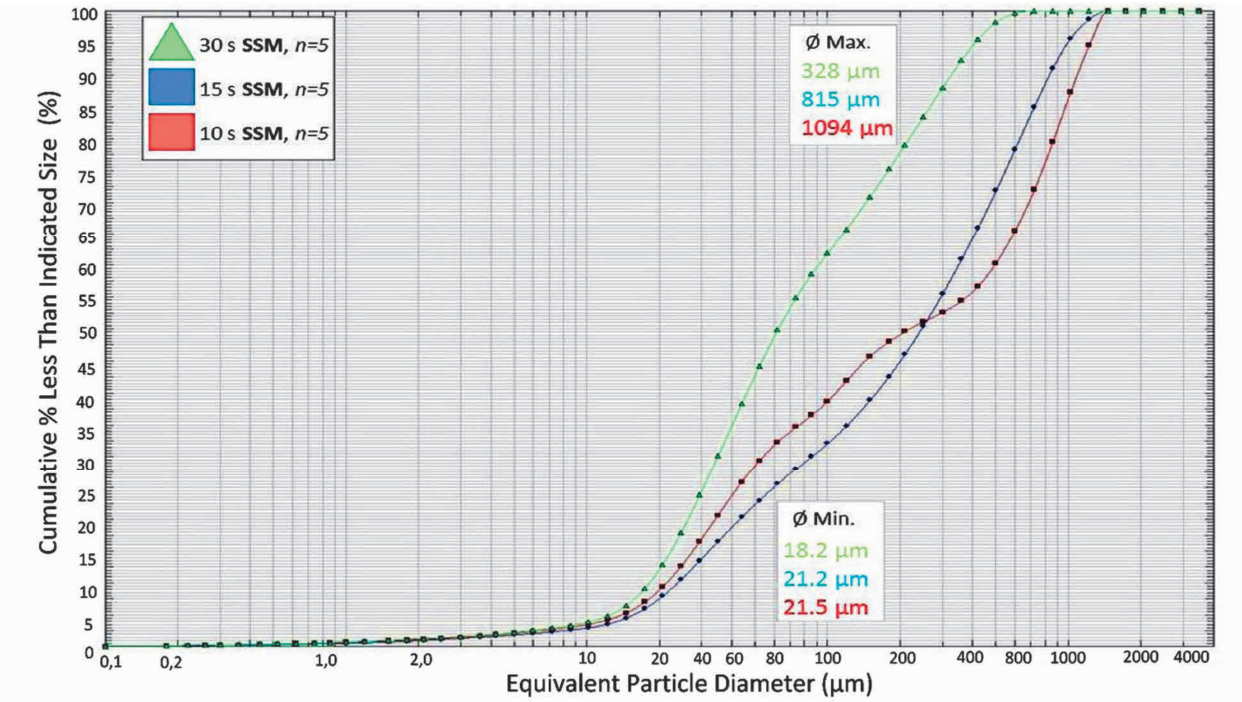


**Figure 3** Results of the particle size analysis in three different milled times of RE-M-B magnetic material recovered from electric motors

The difference in the size of the obtained powder is definitely noticeable despite the small difference of grinding time duration. This is the starting analysis for determination of properties such as the magnetic field strength depending on the particle size.

## 4. CONCLUSION

Quantitative and qualitative characteristics of the output obtained by means of recycling of scrapped electric motors were the basis for the design of the production process of new eco- RE-M-B magnetic materials. On the basis of preliminary research - the study of the chemical composition of powder and particle size distribution of powder - can be concluded that it is possible to reuse magnetically RE-M-B hard magnets. This creates an opportunity to design an innovative recycling technology of magnetic material based on permanent magnets. However, this technology must meet the necessary conditions, which are, from the economic point of view, cost- effectiveness and efficiency.

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## REFERENCES

[1] ELWERT, T., GOLDMANN, D., ROEMER, F., SCHWARZ, S. Recycling of NdFeB magnets from electric drive motors of (hybrid) electric vehicles. *Journal of Sustainable Metallurgy*, 2016, pp. 1-14.

[2] KLIMECKA-TATAR D., BALA H., ŚLUSAREK B., JAGIELSKA-WIADEREK K., The effect of consolidation method on electrochemical corrosion of polymer bonded Nd-Fe-B type magnetic material. *Archives of Metallurgy and Materials*, 2009, vol. 54, no. 1, pp. 247-256.

[3] VAN DER VET, P. E., MARS, N. J. Condorcet query engine. *Journal of the American Society for information science*, 1999, vol. 42, no. 6, pp. 485-492.

[4] SMEJKAL, V. FEM modeling and experimental research of through-thickness strain distribution during hot plate rolling. In *METAL 2014: 23rd International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2014, pp. 289-294.

[5] BINNEMANS, K., JONES, P.T, BLANPAIN B., VAN GERVEN, T., YANG, Y., WALTON, A., BUCHERTF M. Recycling of rare earths: a critical review. *Journal of Cleaner Production*, 2013, vol. 51, pp. 1-22.

[6] BABA, Y., KUBOTA, F., KAMIYA, N., GOTO, M. Recent advances in extraction and separation of rare-earth metals using ionic liquids. *Journal of Chemical Engineering of Japan*, 2011, vol. 44, 679-685.

[7] ITOH, M., MASUDA, M., SUZUKI, S., MACHIDA, K.I., Recycling of rare earth sintered magnets as isotropic bonded magnets by melt-spinning. *Journal of Alloys and Compounds*, 2004, vol. 374, pp. 393-396.

[8] BINNEMANS, K. JONES P.T., BLANPAIN B., VAN GERVEN, T., PONTIKES, Y. Towards zero-waste valorisation of rare-earth-containing industrial process residues: a critical review. *Journal of Cleaner Production*, 2015, vol. 99, 17-38

[9] ZAKOTNIK, M., HARRIS, I. R., & WILLIAMS, A. J. Possible methods of recycling NdFeB-type sintered magnets using the HD/degassing process. *Journal of Alloys and Compounds*, 2008, vol. 450, no. 1, pp. 525-531.

[10] KLIMECKA-TATAR, D., PAWŁOWSKA, G., SOZAŃSKA, M. The effect of powder particle biencapsulation with Ni-P layer on local corrosion bonded Nd-(Fe,Co)-B magnetic material. *Archives of Metallurgy and Materials*, 2015, vol. 60, no. 1, pp. 153-157.

[11] ELWERT, T., GOLDMANN, D. *Entwicklung* eines hydrometallurgischen Recyclingverfahrens für NdFeB-Magnete. Universitätsbibliothek Clausthal, 2015

[12] KLIMECKA-TATAR, D. The powdered magnets technology improvement by biencapsulation method and its effect on mechanical properties. *Manufacturing Technology*, 2014, vol. 14, no. 1, pp. 30-36.

[13] DUPONT, D., BINNEMANS, K. Recycling of rare earths from NdFeB magnets using a combined leaching/extraction system based on the acidity and thermomorphism of the ionic liquid [Hbet][Tf 2 N]. *Green Chemistry*, 2015, vol. 17, no. 4, pp. 2150-2163.

[14] WYSLOCKA, E., ULEWICZ, R. Magnets: history, the current state and the future. In *METAL 2015: 24th International Conference on Metallurgy and Materials*, Ostrava: TANGER, 2015, pp. 1680-1686.

[15] KLIMECKA-TATAR, D., PAWLOWSKA, G., RADOMSKA, K. The effect of Nd12Fe77Co5B6 powder electroless biencapsulation method on atmospheric corrosion of polymer bonded magnetic material. In *METAL 2014: 23rd International Conference on Metallurgy and Materials*. Ostrava: TANGER, 2014, pp. 985-990.

[16] LIU, J., SEPEHRI-AMIN, H., OHKUBO, T., HIOKI, K., HATTORI, A., SCHREFL, T., HONO, K. Effect of Nd content on the microstructure and coercivity of hot-deformed Nd-Fe-B permanent magnets. *Acta Materialia*, 2013, vol. 61, no. 14, pp. 5387-5399

[17] SEPEHRI-AMIN, H., UNE, Y., OHKUBO, T., HONO, K., SAGAWA, M. Microstructure of fine-grained Nd-Fe-B sintered magnets with high coercivity. *Scripta Materialia*, 2011, vol. 65, no. 5, pp. 396-399.

[18] WOODCOCK, T. G., GUTFLEISCH, O. Multi-phase EBSD mapping and local texture analysis in NdFeB sintered magnets. *Acta Materialia*, 2011, vol. 59, no. 3, pp. 1026-1036.

[19] ZHOU, Q., LIU, Z. W., ZHONG, X. C., ZHANG, G. Q. Properties improvement and structural optimization of sintered NdFeB magnets by non-rare earth compound grain boundary diffusion. *Materials and Design*, 2015, vol. 86, pp. 114-120.